

**Electronic Acknowledgement Receipt**
**Ecometal  
Defendants' Exh.**
**001**

Terves v. Yueyang Aerospace (1:19-cv-1611)

exhibitsticker.com

<b>EFS ID:</b>	43181455
<b>Application Number:</b>	90014795
<b>International Application Number:</b>	
<b>Confirmation Number:</b>	2652
<b>Title of Invention:</b>	GALVANICALLY-ACTIVE IN SITU FORMED PARTICLES FOR CONTROLLED RATE DISSOLVING TOOLS
<b>First Named Inventor/Applicant Name:</b>	Brian P. Doud
<b>Customer Number:</b>	30589
<b>Filer:</b>	Marc Alan Brockhaus/Lori Martin
<b>Filer Authorized By:</b>	Marc Alan Brockhaus
<b>Attorney Docket Number:</b>	2189.006
<b>Receipt Date:</b>	06-JUL-2021
<b>Filing Date:</b>	
<b>Time Stamp:</b>	19:58:04
<b>Application Type:</b>	Reexam (Third Party)

**Payment information:**

Submitted with Payment	yes
Payment Type	DA
Payment was successfully received in RAM	\$6300
RAM confirmation Number	E202176J59280435
Deposit Account	602198
Authorized User	Lori Martin

The Director of the USPTO is hereby authorized to charge indicated fees and credit any overpayment as follows:

37 CFR 1.17 (Patent application and reexamination processing fees)

37 CFR 1.19 (Document supply fees)

37 CFR 1.20 (Post Issuance fees)

37 CFR 1.21 (Miscellaneous fees and charges)

**File Listing:**

<b>Document Number</b>	<b>Document Description</b>	<b>File Name</b>	<b>File Size(Bytes)/ Message Digest</b>	<b>Multi Part/.zip</b>	<b>Pages (if appl.)</b>
1	Trans Letter filing of a response in a reexam	2189006_Transmittal.pdf	169393 05368ef59e87530ccf3a893ba24f42235087 5e26	no	4

**Warnings:****Information:**

2	Copy of patent for which reexamination is requested	2189006_653Patent.pdf	5988471 1a05a042deee1ade7a7aff4e4c35925810c8 4c4a	no	20
---	---	-----------------------	---	----	----

**Warnings:****Information:**

3	Information Disclosure Statement (IDS) Form (SB08)	2189006_IDS.pdf	142411 57b1d050a87975f6550eebae57ba0881b39 f13d0	no	2
---	--	-----------------	--	----	---

**Warnings:****Information:**

This is not an USPTO supplied IDS fillable form

4	Foreign Reference	F01_ChineseLanguageXiao.pdf	535925 e90ad1fee65af395ed6f30cec16f845c8b92e d57	no	9
---	-------------------	-----------------------------	--	----	---

**Warnings:****Information:**

5	Foreign Reference	F02_EnglishXiao.pdf	581268 e59b9ab8bcdf696bccdb008e3eb62b8c7e3 2defb	no	18
---	-------------------	---------------------	--	----	----

**Warnings:****Information:**

6	Non Patent Literature	NPL1_16.pdf	11914779 9f51d5e500fb3cd33d2d35803932a08e59c 7ad29	no	106
---	-----------------------	-------------	--	----	-----

**Warnings:**

**Information:**

7	Receipt of Orig. Ex Parte Request by Third Party	2189006_RequestForReExam.pdf	2731961 ef58a64a51d31502aac73b01228aade0d34 2b720	no	300
---	--	------------------------------	---	----	-----

**Warnings:****Information:**

8	Fee Worksheet (SB06)	fee-info.pdf	30252 1caf4d116fff434094ede6b8f77d705924831 408	no	2
---	----------------------	--------------	---	----	---

**Warnings:****Information:**

<b>Total Files Size (in bytes):</b>	22094460
-------------------------------------	----------

**This Acknowledgement Receipt evidences receipt on the noted date by the USPTO of the indicated documents, characterized by the applicant, and including page counts, where applicable. It serves as evidence of receipt similar to a Post Card, as described in MPEP 503.**

**New Applications Under 35 U.S.C. 111**

If a new application is being filed and the application includes the necessary components for a filing date (see 37 CFR 1.53(b)-(d) and MPEP 506), a Filing Receipt (37 CFR 1.54) will be issued in due course and the date shown on this Acknowledgement Receipt will establish the filing date of the application.

**National Stage of an International Application under 35 U.S.C. 371**

If a timely submission to enter the national stage of an international application is compliant with the conditions of 35 U.S.C. 371 and other applicable requirements a Form PCT/DO/EO/903 indicating acceptance of the application as a national stage submission under 35 U.S.C. 371 will be issued in addition to the Filing Receipt, in due course.

**New International Application Filed with the USPTO as a Receiving Office**

If a new international application is being filed and the international application includes the necessary components for an international filing date (see PCT Article 11 and MPEP 1810), a Notification of the International Application Number and of the International Filing Date (Form PCT/RO/105) will be issued in due course, subject to prescriptions concerning national security, and the date shown on this Acknowledgement Receipt will establish the international filing date of the application.

Under the Paperwork Reduction Act of 1995, no persons are required to respond to a collection of information unless it displays a valid OMB control number.

(Also referred to as FORM PTO-1465)

**REQUEST FOR EX PARTE REEXAMINATION TRANSMITTAL FORM**

Address to:

**Mail Stop Ex Parte Reexam  
Commissioner for Patents  
P.O. Box 1450  
Alexandria, VA 22313-1450**

**Attorney Docket No.: 2189.006****Date: 07/06/2021**

1.  This is a request for *ex parte* reexamination pursuant to 37 CFR 1.510 of patent number 10,329,653 B2

issued June 25, 2019. The request is made by:

patent owner.  third party requester.

2.  The name and address of the person requesting reexamination is:

Ecometal, Inc., 35 Owl Ridge Drive, Richmond Hill, Ontario L4S IP8, Canada

3. Requester  asserts small entity status (37 CFR 1.27) or  certifies micro entity status (37 CFR 1.29). Only a patent owner requester can certify micro entity status. Form PTO/SB/15A or B must be attached to certify micro entity status.

4. This request is accompanied by payment of the reexamination fee as set forth in:

37 CFR 1.20(c)(2); or

37 CFR 1.20(c)(1). **In checking this box for payment of the fee set forth in 37 CFR 1.20(c)(1), requester asserts that this request has forty (40) or fewer pages and complies with all other requirements of 37 CFR 1.20(c)(1).**

Payment of the reexamination fee is made by the method set forth below.

a.  A check in the amount of \$ \_\_\_\_\_ is enclosed to cover the reexamination fee;

b.  The Director is hereby authorized to charge the reexamination fee to Deposit Account No. 602198;

c.  Payment by credit card. Form PTO-2038 is attached; or

d.  Payment made via EFS-Web.

In addition, the Director is hereby authorized to charge any fee deficiencies to Deposit Account No. 602198.

5.  Any refund should be made by  check or  credit to Deposit Account No. 602198.  
37 CFR 1.26(c). If payment is made by credit card, refund must be to credit card account.

6.  A copy of the patent to be reexamined having a double column format on one side of a separate paper is enclosed. 37 CFR 1.510(b)(4).

7.  CD-ROM or CD-R in duplicate, Computer Program (Appendix) or large table

Landscape Table on CD

[Page 1 of 3]

This collection of information is required by 37 CFR 1.510. The information is required to obtain or retain a benefit by the public which is to file (and by the USPTO to process) a request for reexamination. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.11 and 1.14. This collection is estimated to take 18 minutes to complete, including gathering, preparing, and submitting the completed application form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, VA 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. **SEND TO: Mail Stop Ex Parte Reexam, Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450.**

If you need assistance in completing the form, call 1-800-PTO-9199 and select option 2.

8.  Nucleotide and/or Amino Acid Sequence Submission  
*If applicable, items a. – c. are required.*
- a.  Computer Readable Form (CRF)
  - b. Specification Sequence Listing on:
    - i.  CD-ROM (2 copies) or CD-R (2 copies) **or**
    - ii.  paper
  - c.  Statements verifying identity of above copies.
9.  A copy of any disclaimer, certificate of correction or reexamination certificate issued in the patent is included.
10.  Reexamination of claim(s) 1–5, 7–9, 11–16, 18–21, 23–50, 52–62, 64, 66, 67, and 69–76 is requested.
11.  A copy of every patent or printed publication relied upon is submitted herewith including a listing thereof on Form PTO/SB/08, PTO-1449, or equivalent.
12.  An English language translation of all necessary and pertinent non-English language patents and/or printed publications is attached.
13.  The attached detailed request includes at least the following items:
  - a. A statement identifying each substantial new question of patentability based on prior patents and printed publications. 37 CFR 1.510(b)(1).
  - b. An identification of every claim for which reexamination is requested, and a detailed explanation of the pertinency and manner of applying the cited art to every claim for which reexamination is requested. 37 CFR 1.510(b)(2).
14.  A proposed amendment is included (only where the patent owner is the requester). 37 CFR 1.510(e).
15.  It is certified that the statutory estoppel provisions of 35 U.S.C. 315(e)(1) or 35 U.S.C. 325(e)(1) do not prohibit requester from filing this *ex parte* reexamination request. 37 CFR 1.510(b)(6).
16. Service
- a.  It is certified that a copy of this request (if filed by other than the patent owner) has been served in its entirety on the patent owner as provided in 37 CFR 1.33(c).
- The name and address of the party served are:  
FAY SHARPE LLP, 1228 Euclid Ave., 5th Floor, The Halle Building, Cleveland, OH 44115
- 
- Brian E. Turung, Esq., ULMER BERNE, 1660 W. 2nd St., Ste. 1100, Cleveland, OH 44113
- 
- Date of Service: July 6, 2021
- OR
- b.  A duplicate copy is enclosed since service on patent owner was not possible. An explanation of the efforts made to serve patent owner **is attached**. See MPEP 2220.

17. Correspondence Address: Direct all communication about the reexamination to:

The address associated with Customer Number: 30589

OR

Firm or Individual Name \_\_\_\_\_  
(at the address identified below)

Address

City	State	Zip
------	-------	-----

Country		
---------	--	--

Telephone	Email
-----------	-------

18.  The patent is currently the subject of the following concurrent proceeding(s):

- a.  Copending reissue Application No. \_\_\_\_\_
- b.  Copending reexamination Control No. \_\_\_\_\_
- c.  Copending Interference No. \_\_\_\_\_
- d.  Copending litigation styled:

Terves, LLC v. Ecometal, Inc. and Mr. Nick Yuan in the U.S. District Court for the Northern District of Ohio, Case No. 1:19-cv-01611-M

**WARNING : Information on this form may become public. Credit card information should not be included on this form. Provide credit card information and authorization on PTO-2038.**

/Marc A. Brockhaus/

July 6, 2021

Authorized Signature

Date

Marc A. Brockhaus

40,923

Typed/Printed Name

Registration No.

For Patent Owner Requester

For Third Party Requester

## Privacy Act Statement

The **Privacy Act of 1974 (P.L. 93-579)** requires that you be given certain information in connection with your submission of the attached form related to a patent application or patent. Accordingly, pursuant to the requirements of the Act, please be advised that: (1) the general authority for the collection of this information is 35 U.S.C. 2(b)(2); (2) furnishing of the information solicited is voluntary; and (3) the principal purpose for which the information is used by the U.S. Patent and Trademark Office is to process and/or examine your submission related to a patent application or patent. If you do not furnish the requested information, the U.S. Patent and Trademark Office may not be able to process and/or examine your submission, which may result in termination of proceedings or abandonment of the application or expiration of the patent.

The information provided by you in this form will be subject to the following routine uses:

1. The information on this form will be treated confidentially to the extent allowed under the Freedom of Information Act (5 U.S.C. 552) and the Privacy Act (5 U.S.C. 552a). Records from this system of records may be disclosed to the Department of Justice to determine whether disclosure of these records is required by the Freedom of Information Act.
2. A record from this system of records may be disclosed, as a routine use, in the course of presenting evidence to a court, magistrate, or administrative tribunal, including disclosures to opposing counsel in the course of settlement negotiations.
3. A record in this system of records may be disclosed, as a routine use, to a Member of Congress submitting a request involving an individual, to whom the record pertains, when the individual has requested assistance from the Member with respect to the subject matter of the record.
4. A record in this system of records may be disclosed, as a routine use, to a contractor of the Agency having need for the information in order to perform a contract. Recipients of information shall be required to comply with the requirements of the Privacy Act of 1974, as amended, pursuant to 5 U.S.C. 552a(m).
5. A record related to an International Application filed under the Patent Cooperation Treaty in this system of records may be disclosed, as a routine use, to the International Bureau of the World Intellectual Property Organization, pursuant to the Patent Cooperation Treaty.
6. A record in this system of records may be disclosed, as a routine use, to another federal agency for purposes of National Security review (35 U.S.C. 181) and for review pursuant to the Atomic Energy Act (42 U.S.C. 218(c)).
7. A record from this system of records may be disclosed, as a routine use, to the Administrator, General Services, or his/her designee, during an inspection of records conducted by GSA as part of that agency's responsibility to recommend improvements in records management practices and programs, under authority of 44 U.S.C. 2904 and 2906. Such disclosure shall be made in accordance with the GSA regulations governing inspection of records for this purpose, and any other relevant (*i.e.*, GSA or Commerce) directive. Such disclosure shall not be used to make determinations about individuals.
8. A record from this system of records may be disclosed, as a routine use, to the public after either publication of the application pursuant to 35 U.S.C. 122(b) or issuance of a patent pursuant to 35 U.S.C. 151. Further, a record may be disclosed, subject to the limitations of 37 CFR 1.14, as a routine use, to the public if the record was filed in an application which became abandoned or in which the proceedings were terminated and which application is referenced by either a published application, an application open to public inspection or an issued patent.
9. A record from this system of records may be disclosed, as a routine use, to a Federal, State, or local law enforcement agency, if the USPTO becomes aware of a violation or potential violation of law or regulation.

## Patent eBusiness

EFS Registered

- [+ Electronic Filing](#)
- [+ Patent Application Information \(PAIR\)](#)
- [+ Patent Ownership](#)
- [+ Fees](#)
- [+ Supplemental Resources & Support](#)

## Patent Information

- [Patent Guidance and General Info](#)
- [+ Codes, Rules & Manuals](#)
- [+ Employee & Office Directories](#)
- [+ Resources & Public Notices](#)

## Patent Searches

- [Patent Official Gazette](#)
- [+ Search Patents & Applications](#)
- [+ Search Biological Sequences](#)
- [+ Copies, Products & Services](#)

## Other

- [Copyrights](#)
- [Trademarks](#)
- [Policy & Law Reports](#)

Registered eFilers <input checked="" type="checkbox"/>	Please Read Announcements	Application Data	Attach Documents	Review Documents	Calculate Fees	Confirm & Submit	Pay Fees	Receipt
--	---------------------------	------------------	------------------	------------------	----------------	------------------	----------	---------

**Review fees and select payment method**

review fees     make payment     fee receipt

**Your Acknowledgement Receipt (evidence of this submission) will be provided upon completion of this payment transaction, or, upon choosing to not pay.**

**This is the application for which you are paying fees.**

EFS ID	43181455
Application Number	90014795
Confirmation Number	2652
Title of Invention	GALVANICALLY-ACTIVE IN SITU FORMED PARTICLES FOR CONTROLLED RATE DISSOLVING TOOLS
First Named Inventor	Brian P. Doud
Customer Number or Correspondence Address	DUNLAP CODDING, P.C. PO BOX 16370 OKLAHOMA CITY OK 73113 US docketing@dunlapcoding.com 405-607-8600 405-607-8686
Filed By	Marc Alan Brockhaus/Lori Martin
Attorney Docket Number	2189.006
Application Type	ex parte reexam

**The following fees will be collected for this application.**

Fee Description	Fee Code	Quantity	Fee Amount	Fee Total
EX PARTE REEXAMINATION (1.510(A)) NON-STREAMLINED	2812	1	6300	6300

Total Payment \$ 6300

**Payment Method**[Read more about the security of your transaction.](#)**New USPTO Payment System** 

Pay using one of the following payment options

which can be stored in your [USPTO.gov Account](#):

- USPTO Deposit Account
- Credit/Debit Card
- Electronic Funds Transfer(EFT)

[Why this change?](#)

[Pay Later, Generate Acknowledgement Receipt](#)

*If you need help:*

- To ask questions about Patent e-Filing, or to suggest improvements to the online system, or report technical problems, please call the Patent Electronic Business Center at (866) 217-9197 (toll free) or send email to [EBC@uspto.gov](mailto:EBC@uspto.gov).
- Send general questions about USPTO programs to the [USPTO Contact Center \(UCC\)](#).
- For general questions regarding a petition, or requirements for filing a petition, contact the Office of Petitions Help Desk at 1 800-786-9199.



[Accessibility](#)

[Privacy Policy](#)

[Terms of Use](#)

[Security](#)

[Emergencies/Security Alerts](#)

[Information Quality Guidelines](#)

[Federal Activities](#)

[Inventory Reform \(FAIR\) Act](#)

[Notification and Federal Employee](#)

[Antidiscrimination and Retaliation \(NoFEAR\) Act](#)

[Budget & Performance](#)

[Freedom of Information Act \(FOIA\)](#)

[Department of](#)

[Commerce NoFEAR Act Report](#)

[Regulations.gov](#)

[STOP!Fakes.gov](#)

[Department of Commerce](#)

[USA.gov](#)

[Strategy Targeting](#)

[Organized Piracy \(STOP!\)](#)

[Careers](#)

[Site Index](#)

[Contact Us](#)

[USPTO Webmaster](#)

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Patent of:  
Doud, et al.

Attorney Docket No. 2189.006

U.S. Patent. No.: 10,329,653

Issued: June 25, 2019

Assignee: Terves, LLC

Mail Stop Ex Parte Reexam  
Central Reexamination Unit Commissioner for Patents  
United States Patent and Trademark Office P.O. Box 1450  
Alexandria, VA 22313-1450

**REQUEST FOR EX PARTE REEXAMINATION OF CLAIMS 1–5, 7–9, 11–16, 18–21, 23–50, 52–62, 64, 66, 67, and 69–76 OF U.S. PATENT NO. 10,329,653**

Ecometal, Inc. (“Requester”) respectfully requests *ex parte* reexamination of claims 1–5, 7–9, 11–16, 18–21, 23–50, 52–62, 64, 66, 67, and 69–76 (“the Challenged Claims”) of U.S. Patent No. 10,329,653 (the “‘653 Patent”).

REFERENCE LIST

<u>Shorthand</u>	<u>Description</u>
'653 Patent	U.S. Patent No. 10,329,653 B2
Medlin Declaration (Exhibit 1)	Declaration of Dana J. Medlin, Ph.D., P.E., FASM Under 37 C.F.R. § 1.68I in Support of Request for <i>Ex Parte</i> Reexamination of U.S. Patent No. 10,329,653
'653 Patent PH	Prosecution History for U.S. Patent No. 10,329,653 B2 (as USPTO has access to this record, Requester is not filing a copy of the prosecution history)
Chinese-Language Xiao	Chinese Patent Publication No. CN103343271A (Xiao) (Chinese-language accompanied by an English language abstract)
Xiao	Chinese Pat. Pub. No. CN 103343271 (Xiao) (Certified English Language Translation)
Hassan	Development of High Strength Magnesium Based Composites Using Elemental Nickel Particulates as Reinforcement. Hassan, S.F., Gupta, M., <i>Journal of Materials Science</i> 37, 2467–2474 (2002).
Abstract Comparison (Exhibit 3)	Abstract Comparison of the English-language abstract and “Abstract of the Disclosures” section of the Certified English-language translation of Chinese Pat. Pub. No. CN 103343271 (Xiao)
'425 Provisional	US Provisional Patent Application 61982425
Hai et al.	Review of Recent Studies in Magnesium Matrix Composters, Hai Zhi Ye, Xing Yang, Liu, <i>Journal of Materials Science</i> 39 (2004)
Hemanth	Fracture Behavior of Cryogenically solidified aluminum-alloy reinforced with Nano-ZrO <sub>2</sub> Metal Matrix Composites (CNMMCs), Joel Hemanth. <i>Journal of Chemical Engineering and Materials Science Vol. 2(8), August 2011</i>

Shivanath	US Patent No. 5,476,632 (Shivanath, et al)
Shaw	Corrosion Resistance of Magnesium Alloys, Barbara A. Shaw, Pennsylvania State University. <i>ASM Handbook, Volume 13A Corrosion: Fundamentals, Testing, and Protection</i>
Al-Fe Phase Diagram	Al-Fe Diagram: Charles H. Henager, Hydrogen Permeation Barrier Coatings, Materials for the Hydrogen Economy, Chapter 8, CRC Press 2007, Editors: R. H. Jones, G. J. Thomas, pp 181-190
Al-Ni Phase Diagram	Al-Ni Diagram: Laszlo J. Kecske et al., Army Research Laboratory ARL-TR-5507, April 2011, ACombustion Synthesis Reaction Behavior of Cold-Rolled Ni/Al and Ti/Al Multilayers. p. 5
Al-Cu Phase Diagram	Al-Cu Diagram: <a href="https://sites.google.com/site/eampotentials/Home/AlCu">https://sites.google.com/site/eampotentials/Home/AlCu</a> downloaded June 20, 2020.
Al-Ag Phase Diagram	Al-Ag Diagram: A. J. McAlister, Bull. Alloy Phase Diagrams, 8(6):526-533, 1987
Cu-Ni Phase Diagram	Cu-Ni Phase Diagram. <i>Teach Yourself Phase Diagrams and Phase Transformations</i> , Professor Mike Ashby, 5th Edition, March 2009 Cambridge
Cu-Ni Chart Phase Diagram	Illustrated portion of Cu-Ni Chart Phase Diagram. <i>Materials Science and Engineering, 6th Edition by Callister and Rethwisch, 2003</i>
Ag-Cu Phase Diagram	Ag-Cu Binary Phase Diagram. <i>ASM Metals Handbook, 8th Edition, Vol. 8, Metallography, Structures and Phase Diagrams, 1973</i>
Mg-Pb Phase Diagram	Mg-Pb Phase Diagram. <i>ASM Metals Handbook, 8th Edition, Vol. 8, Metallography, Structures and Phase Diagrams, 1973</i>

Al-Mg Phase Diagram	Al-Mg Phase Diagram. <i>ASM Metals Handbook, 8th Edition, Vol. 8, Metallography, Structures and Phase Diagrams</i> , 1973
Mg-Ni Phase Diagram	Mg-Ni Diagram. <i>ASM Metals Handbook, 8th Edition, Vol. 8, Metallography, Structures and Phase Diagrams</i> , 1973
Galvanic Series	Jones, D.A.; Principles and Prevention of Corrosion, 2nd Ed., 1996, p. 170 - Galvanic Series Table; 1996. Referred to by POSITAs.
Mass Balance (Exhibit 2)	Mass/Balance. Dana J. Medlin

**I. INTRODUCTION**

Ecometal, Inc. (“Requester”) requests *ex parte* reexamination under 35 U.S.C. § 302 and 37 C.F.R. § 1.501 of claims 1–5, 7–9, 11–16, 18–21, 23–50, 52–62, 64, 66, 67, and 69–76 of U.S. Patent No. 10,329,653 (“the ’653 Patent” and “the ’653 Patent Claims”). This Request sets forth grounds pointing out each substantial new question of patentability based on prior patents and printed publications, which are likely to prevail based on the accompanying evidence, and as such, this Request should be granted and the ’653 Patent Claims cancelled.

**II. PAYMENT OF FEES PURSUANT TO 37 C.F.R. § 1.510(a)**

Per 37 C.F.R. § 1.510(a), the Office is authorized to charge \$6,300.00 and any additional fees to Deposit Account No. 602198.

**III. RELATED PROCEEDINGS**

The ’653 Patent is the subject of a patent infringement lawsuit brought by the Patent Owner Terves, LLC against Requester and Mr. Nick Yuan in the U.S. District Court for the Northern District of Ohio, Case No. 1:19-cv-01611-M (“Related Litigation”). The ’653 Patent was also the subject a petition for *inter partes* review initiated by Requester on September 11, 2020, IPR2020-1620. On March 1, 2021, the Patent Trial and Appeal Board issued a decision (Paper 9) denying institution based on its conclusion that Requester filed its petition for *inter partes* review more than one year after being served, pursuant to the Hague Convention, with a complaint alleging infringement of the ’653 Patent.

**IV. IDENTIFICATION OF CLAIMS FOR WHICH REXAMINATION IS REQUESTED (37 C.F.R. 1.510(b)(2))**

Requester seeks *ex parte* reexamination of claims 1–5, 7–9, 11–16, 18–21, 23–50, 52–62, 64, 66, 67, and 69–76 (“’653 Patent Claims”).

V. **REQUESTER IS NOT ESTOPPED UNDER 35 U.S.C. 315(e)(1) or 35 U.S.C. 325(e)(1) FROM FILING THIS REQUEST (37 C.F.R. § 1.510(b)(6))**

Requester certifies they are not estopped from filing this Request under 35 U.S.C. § 315(e)(1) or 35 U.S.C. § 325(e)(2). Requester is not a Petitioner in, nor is any real party in interest or privy of Requester in, an *inter partes* review (35 U.S.C. § 315(e)(1)) or a post-grant review (35 U.S.C. § 325((e)(1)) of the ‘653 Patent that has resulted in a final written decision.

**1. OVERVIEW OF THE ‘653 PATENT**

**A. Effective Filing Date of the ‘653 Patent**

The ‘653 Patent issued on June 25, 2019, from U.S. Patent Application No. 15/641,439, filed July 5, 2017. The ‘653 Patent claims priority to U.S. Provisional Application No. 61/981,425 (Ex. 1009), which was filed April 18, 2014 the earliest effective filing date of the ‘653 Patent.

**B. The Specification**

The ‘653 Patent’s purported invention is a “magnesium composite for use as a dissolvable component in oil drilling” such as “a ball or other tool component in a well drilling or completion operation such as, but not limited to, a component that is seated in a hydraulic operation that can be dissolved away after use so that no drilling or removal of the component is necessary.” Ex. 1001, at 1:48–59.<sup>1</sup> The ‘653 Patent describes the magnesium composite as used to form a “castable, moldable, or extrudable component.” *Id.* at 2:19–20.

The inventive magnesium composite includes “at least 50 wt.% magnesium” with “one or more additives added to a magnesium or magnesium alloy to form the novel magnesium composite.” *Id.* at 2:20–24. The “one or more additives can be selected and used in quantities so that galvanically-active intermetallic or insoluble precipitates form in the magnesium or

---

<sup>1</sup> The notation N:XX–YY refers to column (or page) number N, and lines XX to YY of a reference.

magnesium alloy while the magnesium or magnesium alloy is in a molten state and/or during the cooling of the melt.” *Id.* at 2:24-29.

The ’653 Patent further describes “one or more additives can be added to the molten magnesium or magnesium alloy at a temperature that is greater than the melting point of the one or more additives.” *Id.* at 2:53–56. “Generally, the temperature of the molten magnesium or magnesium alloy is at least about 10°C less than the melting point of the additive added to the molten magnesium or magnesium alloy during the addition and mixing process, typically at least about 100° C less than the melting point of the additive added to the molten magnesium or magnesium alloy during the addition and mixing process.” *Id.* at 3:2–8. The process may “achieve a specific galvanic corrosion rate in the entire magnesium composite and/or along the grain boundaries of the magnesium composite.” *Id.* at 3:16–18.

The ’653 Patent discloses the “invention adopts a feature that is usually a negative in traditional casting practices wherein a particle is formed during the melt processing that corrodes the alloy when exposed to conductive fields and is imbedded in eutectic phases, the grain boundaries, and/or even within grains with precipitation hardening.” *Id.* at 3:19–24. Adoption of this previously-known “negative” feature “result[ed] in the ability to control where the galvanically-active phases are located in the final casting, as well as the surface area ratio of the in situ phase to the matrix phase, which enables the use of lower cathode phase loadings as compared to a powder metallurgical or alloyed composite to achieve the same dissolution rates;” thus, the in situ formed galvanic additives can be used to improve certain mechanical properties of the magnesium composite, including ductility, tensile strength, and/or shear strength. *Id.* at 3:24–30.

**V. FIELD OF TECHNOLOGY**

The field of endeavor of the '653 Patent is changing the physical structure of non-ferrous metals or alloys by heat treatment or by hot or cold working of magnesium or alloys based thereon.

For a detailed discussion of the background of the '653 Patent and Field of Technology, see the Medlin Declaration at ¶¶ 44–77.

## **VI. LEVEL OF ORDINARY SKILL IN THE PERTINENT ART**

A POSITA at the time of filing of the '653 Patent, i.e., April 18, 2014, had at least a bachelor's degree in metallurgical, materials, or mechanical engineering, or the equivalent, and at least five years of experience in metallurgical and materials engineering, or an equivalent amount of relevant work and/or educational experience.

## **VII. STATEMENT POINTING OUT EACH SUBSTANTIAL NEW QUESTION OF PATENTABILITY (37 C.F.R. § 1.510(b)(1))**

### *A. Prior Art Patents and Printed Publications Relied on*

Requester relies on:

1. Certified English-Language Translation of Chinese Patent Publication No. CN 103343271 to Xiao *et al.* ("Xiao").
2. Development of High Strength Magnesium Based Composites Using Elemental Nickel Particulates as Reinforcement. Hassan, S.F., and Gupta, M., *Journal of Materials Science* 37, 2467–2474 (2002) ("Hassan").

A Reference List identifying the other exhibits relied upon in this request or the accompanying expert declaration is included, at p. 2, *supra*.

Xiao and Hassan were each published before the earliest claimed priority date of the '653 Patent Claims – April 18, 2014<sup>2</sup> (the "Priority Date"); thus, each is prior art to the '653 Patent Claims under at least AIA 35 U.S.C. § 102 and 35 U.S.C. § 103.

---

<sup>2</sup> **Effective Filing Date of the '653 Patent.** The '653 Patent issued on June 25, 2019, from U.S. Patent Application No. 15/641,439, filed July 5, 2017. The '653 Patent claims priority to U.S. Provisional Application No. 61/981,425 (Ex. 1009), which was filed April 18, 2014 the earliest effective filing date of the '653 Patent.

Specifically, Xiao was filed on July 8, 2013 and published October 9, 2013. Xiao qualifies as a printed publication and prior art under 35 U.S.C. § 102(a)(1). Hassan qualifies as a “printed publication” because it was “disseminated or otherwise made available to the extent that persons interested and ordinarily skilled in the subject matter or art exercising reasonable diligence [could] locate it” before the critical date, i.e. April 18, 2014. *SRI Int'l, Inc. v. Internet Sec. Sys.*, 511 F.3d 1186, 1194 (Fed. Cir. 2008); see also, *Voter Verified, Inc. v. Premier Election Solutions, Inc.*, 698 F.3d 1374, 1380 (Fed. Cir. 2012) (quoting *In re Cronyn*, 890 F.2d 1158, 1160 (Fed. Cir. 1989)).

Hassan was published in the June 2002 issue of the Journal of Materials Science, as: Hassan, S.F., Gupta, M. Development of high strength magnesium-based composites using elemental nickel particulates as reinforcement. *Journal of Materials Science* 37, 2467–2474 (2002). Hassan, cover page. Journal of Material Science was and is reputable, widely known, and readily accessible to POSITAs. Hassan bears a copyright date of 2002. *Id.*, *LG Elec., Inc. v. Advanced Micro Devices, Inc.*, IPR2015-00329, Paper 13, at 12 (PTAB Jul. 10, 2015) (copyright date is prima facie evidence of publication). Hassan further has conventional indicia of its accessibility prior to the critical date. See *Provepharm Inc. v. Wista Labs. Ltd.*, 2018 WL 3326805, at 7 (P.T.A.B. July 6, 2018). (If the “conventional markers” support a finding that the reference is prior art, then, “absent evidence to the contrary, [the Board] will have no reason to suspect otherwise.”). Hassan was marked “Received 14 June 2001 and accepted 12 February 2002” by Kluwer Academic Publishers. Hassan, at 8. Volume 37, which contained Hassan, was assigned an ISSN number (0022-2461)—indicating it is published and available for public access. Moreover, Hassan was cited by POSITAs prior to April 18, 2014. (See, e.g., Hai, at 18; Hemanth, at 11.). Thus, Hassan qualifies as a printed publication and prior art under 35 U.S.C. § 102(a).

Section IX’s Detailed Explanation includes several references that, though not necessary

to render the '653 Patent Claims anticipated/obvious and therefore not listed in the relied-on references above, provide evidentiary support for a POSITA's understandings of and motivations to combine the relied-on references. These references include:

- Formation of Magnesium Metal Matrix Composites Al203p/Az91D and Their Mechanical Properties After Heat Treatment, Lin et al. (showing known magnesium alloy represented by Mg-Al-Zn).

Should the office desire, this reference may be considered relied-on and part of the Proposed Ground (discussed below).

*B. At least Xiao and Hassan raise Substantial New Questions of Patentability (SNQs")*

a. Prosecution History of the '653 Patent

The '653 Patent was filed on July 5, 2017. On September 20, 2017, Patent Owner submitted an IDS disclosing eighty-eight references including Chinese Patent Publication No. CN103343271A (in Chinese) accompanied by an English language abstract ("Chinese-language Xiao"). The submitted references were considered by the examiner.

Following a Restriction Requirement and Patent Owner's response thereto, the Office issued a November 21, 2018 Office Action, rejecting claims 22, 24, 25, 29–32, 63, 64, 67–70, 75, 75, and 81–86 as anticipated by or, in the alternative, obvious over JP2013-019030 (Tashiharu referred to hereinafter as JP '030), and claims 66 and 72 as obvious over JP'030.

In response to this Office Action, Patent Owner responded to the rejections by claiming that they were moot in view of the amendments and, for a new claim 87, by arguing it that the CaMgSi phase in the magnesium alloy of JP'030 is likely not galvanically-active and would not corrode in the presence of a KCl water mixture because it discloses a magnesium alloy which is usable in "transport-airplane machines" and other materials of an automobile, an airplane and a rail car, a machine part, and a robot part article and that "***a person having ordinary skill in the art***

*would understand that those magnesium alloys that are used in automobiles either improve corrosion resistance or are corrosion resistant.”* (emphasis added).

A Notice of Allowance issued on March 27, 2019, finding the claims allowable because “the closest prior art of record,” JP2013-019030 (Tashiharu JP’030) does not teach or suggest the additive(s) including copper, nickel, iron, or cobalt, or mixtures thereof.

b. SNQ Raised by Xiao

Requester asserts that a full English translation of Xiao teaches all features of claims 1–5, 9, 11, 29–32, 37–50, 52–54, 56–62, 64, 66, 67, 69, 70, and 72–73 and are thereby anticipated by Xiao, and claims 8, 25–28, 33–36, 55, and 74–78 are at least made obvious, by Xiao in combination with the prior art magnesium also disclosed in Xiao.

A Chinese language version of Xiao (with an English language abstract) was made of record in the ’653 Patent in an IDS submitted by the Patent Owner dated September 20, 2017. The Information Disclosure Statement disclosed twenty-eight (28) U.S. Patents, thirty-two (34) U.S. Patent publications, fifteen (15) Foreign Patent Documents, and thirteen (13) Non-Patent Literature Documents. *Id.* at 201–209. The Foreign Patent Documents included Chinese Patent Publication No. CN103343271A (in Chinese) accompanied by an English language abstract (hereinafter referred to as “Chinese-language Xiao”).

An English-language abstract of Xiao was provided to the Examiner. Using MS Word, Requester compared that English-language abstract to the “Abstract of the Disclosures” section of the Certified translation. Exhibit 3. The comparison shows that the abstracts would have been considered practically identical by a POSITA. Medlin Decl. Exhibit 2 at ¶ 84. However, both Abstracts failed to disclose the important subject matter of the Xiao reference: the Abstracts say nothing about Xiao’s disclosures of galvanic-activity, corrosion or accelerating the dissolution.

Rather, the Abstracts say that “the alloy decomposition performance exceeds that of a traditional cast magnesium alloy.” *Id.* This portion of the final sentence of the Xiao Abstract likely led the Examiner to incorrectly believe that Xiao—like much of the magnesium alloy prior art (see, e.g., the ’653 Patent, at 3:19-24) and JP’030 (considered the closest reference by the Examiner)—disclosed nothing more than “magnesium alloys [that] either improve corrosion resistance or are corrosion resistant.” ’653 PH, at 606–07. *In re Swanson*, 540 F.3d 1368, 1378 (Fed. Cir. 2008), clarified that a “substantial new question of patentability” refers to a question which has never been considered by the USPTO. The proper question is not whether a reference has been considered, but whether the particular question of patentability presented by the reference in reexamination was previously evaluated by the USPTO, taking into account the context and scope of the consideration. *Id.* at 1379–80. As such, the question of patentability presented by the English translation of the Xiao reference was certainly not evaluated by the USPTO during prosecution of the ’653 patent because the USPTO did not have an English translation of the germane portion of the Xiao reference.

As detailed below in Section IX, the Xiao reference, when reviewed in its entirety, anticipates claims 1–5, 9, 11, 29–32, 37–50, 52–54, 56–62, 64, 66, 67, 69, 70, and 72–73 of the ’653 patent and also renders claims 7–8, 12–16, 18–21, 23–28, 33–36, 55, 71, and 74–78 of the ’653 patent obvious in view of Hassan. As such, the Xiao reference, in its entirety, presents a substantial new question of patentability.

c. SNQ Raised by Hassan

Requester also asserts that Hassan in combination with Xiao raises a substantial new question of patentability as claims 7, 12–16, 18–21, 23–24, and 71 are obvious over Xiao in

combination with Hassan as discussed below. The prosecution history of the '653 patent shows that Hassan was not considered by the Examiner during the prosecution of the '653 Patent.

Hassan (like Xiao) discloses “[m]agnesium based materials due to their inherently low density and ensuing potential to exhibit high specific mechanical properties are actively sought for weight-critical structural application.” Hassan at 2467. Hassan discloses the “synthesis of monolithic and nickel reinforced magnesium composites (Mg/Nip),” including “superheating the magnesium turning with reinforcement particulates (placed in multi-layer sandwich form) to 750°C” and stirring the “molten slurry.” *Id.* at 2468.

Hassan describes its study as using “magnesium turnings . . . as the base material and elemental nickel particulates of 99.9% purity (Johnson Matthey, MA, USA) with an average size of  $29\pm19 \mu\text{m}$  were used as reinforcement phase.” *Id.* at 2468. Figure 1 of Hassan demonstrates “representative SEM micrographs showing distribution of nickel and its reaction product in: (a) Mg/7.3 Nip, (b) Mg/14.0Nip and (c) Mg/24.9Nip matrices.”

Further, Hassan also discloses that the deposited magnesium ingots may then be subjected to secondary processing. For example, Hassan teaches that the nickel-reinforced magnesium ingots are machined and hot extruded to obtain rods of 8 mm diameter. *Id.* Analysis of the composite of Hassan “showed the reduction in the size of elemental nickel particulates in the extruded composites” (see Table 1 of Hassan). In other words, the extrusion in Hassan resulted in a reduction of particulate size of the magnesium composite.

Hassan therefore contains disclosures that some of the additive material may remain “unreacted” with the molten magnesium; a reduction in particulate size of the additive material; and that the magnesium ingots may be subjected to secondary processing; none of these disclosures were considered by the Examiner during prosecution of the '653 Patent. As such, Hassan, when

viewed in combination with Xiao, raises a substantial new question of patentability with respect to whether or not 7, 12–16, 18–21, 23–24, and 71 are obvious.

*C. No Other Proceeding Exists*

According to the MPEP § 2242(I), a substantial new question of patentability is new so long as, in relevant part, it has not been “decided in an earlier concluded examination or review of the patent by the Office” such as “in an earlier concluded trial by the Patent Trial and Appeal Board.” It is relevant that no other proceeding has been instituted regarding the ’653 Patent; therefore, a substantial new question of patentability is at issue.

**VIII. OVERVIEW OF INVALIDATING PRIOR ART**

**A. The English Translation version of Xiao**

Xiao, entitled “Light and pressure-proof fast-decomposed cast magnesium alloy,” discloses a “cast magnesium alloy, which can be used as a tripping ball material for a multi-stage sliding sleeve staged-fracturing technique.” Xiao, at ¶ 0001. Xiao explains “[i]n recent years, the United States has made breakthroughs in the technologies of shale gas exploration and development, and the output has grown rapidly.” “After the completion of all rock fracturing, it is necessary to depressurize the well tubing to facilitate the later production of the oil and gas well. In the past, the conventional method is to use the low-layer pressure to push the ball back to the well opening, or use a drilling tool to drill the sliding sleeve ball seat and the tripping ball, so as to destroy them.” *Id.* at ¶ 0002.

Xiao identifies the need for “the development of a tripping ball that can withstand the high pressure of fracturing construction and the high temperature at the bottom of the well, and has the feature of self-decomposition in the fluid environment of a well, can effectively reduce the construction risk and improve the construction efficiency.” *Id.*

Xiao sought to meet this need by proposing a “method for preparing a light and pressure-proof fast-decomposed cast magnesium alloy by means of optimizing the alloy composition design using a self-made flux containing a rare earth element and adopting a smelting casting method.” *Id.* at ¶ 0004. Specifically, Xiao discloses a “magnesium alloy with a high aluminum content (13 to 25% by weight) and a high zinc content (2 to 10% by weight), and further adds elements of Fe, Cu, Ni and Ag which can enhance the corrosion performance of the magnesium alloy, and at the same time adds Zr, Ti element as grain refiners to improve the compressive strength of the formed material, in which the purpose of adding the high content of aluminum is to produce a large amount of cathode phase, the  $\beta$  ( $Mg_{17}Al_{12}$ ) phase and eutectic phase on the grain boundary of the magnesium alloy, and the magnesium matrix  $\alpha$  phase functions as an anode phase, so that the matrix and the grain boundary of the magnesium alloy form a large amount of micro-batteries, which [micro-batteries] greatly accelerate the corrosion decomposition of magnesium alloy.” *Id.* at ¶ 0026.

Discussed in-depth, below, Xiao also discloses specific dissolution/decomposition rates of its novel magnesium composites. Specifically, Xiao includes the following table, which discloses the various dissolution/decomposition rates:

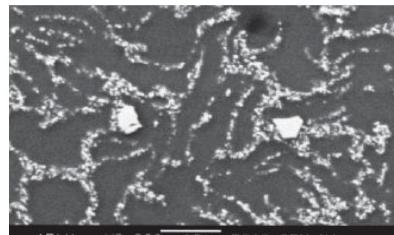
	Room temperature tensile strength $\sigma_b$ (MPa)	Decomposition rate at 70 °C in 3% KCl solution (g.cm <sup>-2</sup> .h <sup>-1</sup> )	Decomposition rate at 93 °C in 3% KCl solution (g.cm <sup>-2</sup> .h <sup>-1</sup> )
Comparative example 1	232	0.00026	0.0005
Example 1	360	0.035	0.074
Example 2	385	0.015	0.045
Example 3	410	0.013	0.036
Example 4	375	0.034	0.058
Example 5	392	0.025	0.048
Example 6	365	0.021	0.063
Example 7	387	0.036	0.057

*Id.* at ¶ 0064.

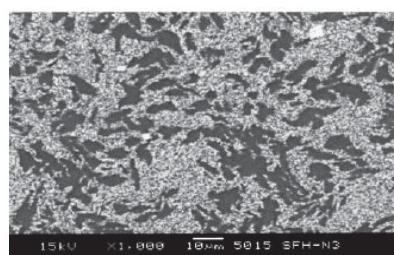
## B. Hassan

Prior art Hassan (introduced above) recognized that “[m]agnesium based materials due to their inherently low density and ensuing potential to exhibit high specific mechanical properties are actively sought for weight-critical structural application.” Ex. 1007 at 2467. Hassan discloses the “synthesis of monolithic and nickel reinforced magnesium composites (Mg/Nip),” including “superheating the magnesium turning with reinforcement particulates (placed in multi-layer sandwich form) to 750°C” and stirring the “molten slurry.” *Id.* at 2468.

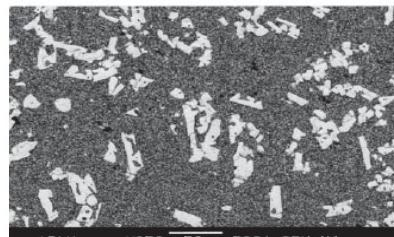
Hassan describes its study as using “magnesium turnings . . . as the base material and elemental nickel particulates of 99.9% purity (Johnson Matthey, MA, USA) with an average size of 29±19 µm were used as reinforcement phase.” *Id.* at 2468. Figure 1 of Hassan demonstrates “representative SEM micrographs showing distribution of nickel and its reaction product in: (a) Mg/7.3 Nip, (b) Mg/14.0Nip and (c) Mg/24.9Nip matrices.”



(a)



(b)



(c)

Hassan also discloses the deposited magnesium ingots may then be subjected to secondary processing. For example, Hassan teaches that the nickel-reinforced magnesium ingots are machined and hot extruded to obtain rods of 8 mm diameter. *Id.* Analysis of the composite of Hassan “showed the reduction in the size of elemental nickel particulates in the extruded composites” (see Table 1 of Hassan). In other words, the extrusion in Hassan resulted in a reduction of particulate size of the magnesium composite. Hassan was not considered by the patent examiner during the prosecution of the ‘653 Patent.

**IX. DETAILED EXPLANATION OF THE PERTINENCY AND MANNER OF APPLYING THE CITED PRIOR ART TO THE '653 PATENT CLAIMS (37 C.F.R. § 1.510(b)(1)).**

**A. Ground 1: Claims 1–5, 9, 11, 29–32, 37–50, 52–54, 56–62, 64, 66, 67, 69, 70, and 72–73 are Anticipated by Xiao**

**i. Anticipation by Xiao**

**1. Anticipation**

“When, as by a recitation of ranges or otherwise, a claim covers several compositions, the claim is ‘anticipated’ if one of them is in the prior art.” *Titanium Metals Corp. v. Banner*, 778 F.2d 775, 227 USPQ 773 (Fed. Cir. 1985).

When the prior art discloses a range which touches or overlaps the claimed range, but no specific examples falling within the claimed range are disclosed, the reference anticipates the claims if the claimed subject matter is disclosed in the reference with "sufficient specificity." See MPEP 2131.03.

For a disclosure to be implicit or inherent it must be a necessary feature or result of an embodiment sufficiently described and enabled in the prior art reference. (In other words, that a certain result or characteristic may occur or be present in the prior art reference is not enough; that certain result or characteristic must always occur (or be present) for the result or characteristic to be implicitly or inherently disclosed.) However, it is not necessary that POSITA would have recognized that implicit or inherent disclosure at the time of invention. See, e.g., *MEHL/Biophile Int'l Corp. v. Milgram*, 192 F.3d 1362, 1365 (Fed. Cir. 1999).

"To serve as an anticipation when the reference is silent about the asserted **inherent** characteristic, such gap in the reference may be filled with recourse to extrinsic evidence. Such evidence must make clear that the missing descriptive matter is necessarily present in the thing described in the reference, and that it would be so recognized by persons of ordinary skill."

*Continental Can Co. USA v. Monsanto Co.*, 948 F.2d 1264, 1268, 20 USPQ2d 1746, 1749-50 (Fed. Cir. 1991) ("this modest flexibility in the rule that 'anticipation' requires that every element of the claims appear in a single reference accommodates situations in which the common knowledge of technologists is not recorded in the reference; that is, where technological facts are known to those in the field of the invention, albeit not known to judges."). Note that as long as there is evidence of record establishing inherency, failure of those skilled in the art to contemporaneously recognize an **inherent** property, function or ingredient of a prior art reference does not preclude a finding of anticipation. *Atlas Powder Co. v. IRECO, Inc.*, 190 F.3d 1342, 1349, 51 USPQ2d 1943, 1948 (Fed. Cir. 1999) (Two prior art references disclosed blasting compositions containing water-in-oil emulsions with identical ingredients to those claimed, in overlapping ranges with the claimed composition. The only element of the claims arguably not present in the prior art compositions was "sufficient aeration . . . entrapped to enhance sensitivity to a substantial degree." The Federal Circuit found that the emulsions described in both references would inevitably and inherently have "sufficient aeration" to sensitize the compound in the claimed ranges based on the evidence of record (including test data and expert testimony). This finding of inherency was not defeated by the fact that one of the references taught away from air entrapment or purposeful aeration.).

If the claimed composition is identical to a composition disclosed by a prior art reference, it does not matter that the applicant recognized additional properties of that composition. See *Titanium Metals Corp. of America v. Banner*, 778 F.2d at 782. See also *In re Spada*, 911 F.2d 705 (Fed. Cir. 1990) ("[t]he discovery of a new property or use of a previously known composition, even when that property and use are unobvious from the prior art, cannot impart patentability to claims to the known composition").

In *Titanium Metals*, the Federal Circuit found that the prior art anticipated the claims at issue despite the alleged newly-discovered property of the claimed composition. The claims were directed to a titanium base alloy containing certain percentages of particular metals and having good corrosion resistance. The Federal Circuit found the claims anticipated by a Russian article, which disclosed an alloy having percentages of metals falling within those claimed. The applicants argued that the prior art did not disclose the newly-discovered corrosion resistance property. The court rejected this argument. It reiterated the well-established rule that the recitation of a newly-discovered property in a claim does not render novel an otherwise known composition where that property is inherent in the composition. *Titanium Metals Corp. of America v. Banner*, 778 F.2d at 782.

As stated in *Scripps Clinic & Research Foundation v. Genentech, Inc.* 927 F.2d 1565, 1567–77 (Fed. Cir. 1991):

It is sometimes appropriate to consider extrinsic evidence to explain the disclosure of a reference. Such factual elaboration is necessarily of limited scope and probative value, for a finding of anticipation requires that all aspects of the claimed invention were already described in a single reference: a finding that is not supportable if it is necessary to prove facts beyond those disclosed in the reference in order to meet the claim limitations. The role of extrinsic evidence is to educate the decision-maker to what the reference meant to persons of ordinary skill in the field of the invention, not to fill gaps in the reference. See *Studiengesellschaft Kohle, mbH v. Dart Industries, Inc.*, 726 F.2d 724, 727, 220 USPQ 841, 842 (Fed.Cir.1984) (although additional references may serve to reveal what a reference would have meant to a person of ordinary skill, it is error to build “anticipation” on a combination of these references). If it is necessary to reach beyond the boundaries of a single reference to provide missing disclosure of the claimed invention, the proper ground is not § 102 anticipation, but § 103 obviousness.

**1. Independent Claims 1, 29, 37, 41, 45, 49, and 73 and Dependent Claims 2–5, 9, 11, 30–32, 38–40, 42–44, 46–48, 50, 52–54, 56–62, 64, 66, 67, 69, 70, and 72 are Anticipated by Xiao**

Pursuant to 37 C.F.R. § 1.510 and MPEP § 2214, Requester provides the following charts depicting where claim elements are present in the prior art. The grounds, below, and the concurrently-submitted Declaration of Dr. Dana J. Medlin provide a detailed explanation of the pertinency and manner of applying the prior art to each element of the Challenged Claims. For each challenged dependent claim, if not explicitly stated, Requester incorporates its analysis for each claim (independent or dependent) from which the challenged dependent claim depends:

<u>'653 Patent</u> <u>Claims/Elements</u>	<u>Anticipated by Disclosures of CN 103343271 A</u> <u>Xiao et al. (“Xiao”)</u>
<b>Independent Claim 1</b>	<b>Disclosure of Claim Element in Xiao</b>
1. A magnesium composite that includes in situ precipitation of galvanically-active intermetallic phases to enable controlled dissolution of said magnesium composite,	<p>Xiao discloses a “magnesium composite”, i.e., “magnesium alloy...and further adds elements of Fe, Cu, Ni, and Ag which can enhance the corrosion performance of the magnesium alloy...” ¶ 0026, ll. 1-3.</p> <p>Xiao also discloses a magnesium composite that includes in situ precipitation of galvanically-active intermetallic phases to enable controlled dissolution of said magnesium composite. Specifically, Xiao discloses a novel magnesium alloy (i.e., magnesium composite) with high aluminum and zinc content further adding elements Fe, Cu, Ni and Ag which can form intermetallics and enhance the corrosion performance of the magnesium alloy. Xiao explains the corrosion mechanism within a magnesium alloy in terms of a cathode phase, which Xiao explains includes “the <math>\beta</math> (Mg<sub>17</sub>Al<sub>12</sub>) phase and eutectic phase on the grain boundary of the magnesium alloy,” and “the</p>

	<p>magnesium matrix <math>\alpha</math> phase which functions as an anode phase, so that the matrix and the grain boundary of the magnesium alloy form a large amount of micro-batteries, which [micro-batteries] greatly accelerate the corrosion decomposition of magnesium alloy.” ¶ 0026.</p> <p>A POSITA in August 2014 would have understood this disclosure of “intermetallic composite micro-particles” to be a reference to intermetallic precipitate.</p> <p>A POSITA reading the Xiao reference in April 2014 would have recognized that the precipitation of galvanically-active intermetallic phases (“micro-batteries”) are naturally and necessarily generated by the <i>in situ</i> reaction between magnesium and any and all of the disclosed metal additives forming <i>in situ</i> precipitate. Thus, this claim element was disclosed by Xiao.</p>
said magnesium composite comprising a mixture of magnesium or a magnesium alloy and an additive material,	Xiao discloses “first loading pure magnesium and pure aluminum into a smelting furnace . . . next loading pure zinc and an [sic] intermediate alloys of trace element components into a resulting magnesium-aluminum alloy melt after melting.” ¶ 0022. This is a “magnesium composite comprising a mixture of magnesium or a magnesium alloy.” Xiao also discloses “an additive material”, i.e., Xiao discloses that “Al-Fe . . . Al-Ni . . . Al-Cu . . . Al- Ag . . . Al-Zr . . . and . . . Al-Ti intermediate alloy[s], are heated to dry and then added to the magnesium-aluminum alloy melt.” Id. at ¶ 0023. An additive material is a material that is added. The addition of these intermediate alloys to the melted magnesium alloy necessarily results in a mixture of the additive material and magnesium alloy.

<p>said additive material having a greater melting point temperature than a solidus temperature of said magnesium,</p>	<p>Using a typical phase diagram, a POSITA in April 2014 would have readily determined that “said additive material having a greater melting point temperature than a solidus temperature of said magnesium.” The solidus temperature of magnesium is the temperature at which a metal first begins to melt during heating and not necessarily when the metal is completely liquidus.</p> <p>A POSITA would also use this phase diagram to find the solidus temperature of the magnesium-aluminum alloy disclosed in Xiao to be in a range of 650°C for pure magnesium and down to 437°C for aluminum additions in the range of 13 wt% to 25% and even up to 40 wt%.</p> <p>The POSITA using typical phase diagrams for each of the additive materials (here the Al-Fe, Al-Ni, Al-Cu, Al-Ag, Al-Zr, Al-Ti “intermediate alloys” disclosed in Xiao) would be able to readily determine the melting point temperature (i.e. “temperature at which liquid is first formed” for each intermediate alloy. (¶ 0023). For example, the Al-Fe phase diagram (Ex. 1205) shows a variable melting point temperature ranging from 1538°C (pure iron) down to 655°C for a eutectic temperature between about 63 wt% aluminum to near 99 wt% aluminum. The Al-Ni phase diagram (Ex. 1206) shows a variable melting point temperature that ranges from 1640°C down to 640°C depending upon the alloy content. The Al-Cu (Ex. 1207) phase diagram shows a variable melting point temperature ranging from 548°C up to 1084°C depending upon the alloy mixture. A POSITA in April 2014 would have readily determined the specific melting temperature of a specific alloy mixture based on these phase diagrams.</p>
--	--

	<p>Xiao discloses a variety of Al-Fe, Al-Ni, Al-Cu, Al-Ag, Al-Zr, Al-Ti alloy mixtures with a variety of constituencies. For instance, Xiao specifically teaches in Example 7 that:</p> <p>The composition of the alloy and the respective percentages by weight are as follows: 25% Al-10% Zn-1% Fe-0.5% Ni-0.1% Cu- 0.5% Ti-0.05% Zr, and the remainder is Mg.</p> <p>¶ 0058-59. Xiao teaches that the magnesium alloy of Example 7, as a whole, ends up with the maximum amount of aluminum (i.e. 25 wt%) taught as being within the invention of Xiao (see, ¶ 0026). Xiao specifically discloses—with respect to Example 7—that portions of that aluminum content are added into a magnesium-aluminum melt as an Al-Fe intermediate alloy, an Al-Ni intermediate alloy, an Al-Cu intermediate alloy, an Al-Ag intermediate alloy, an Al-Zr intermediate alloy, and an Al-Ti intermediate alloy. ¶ 0060. By converting weight percentages to grams and calculating mass balances, a POSITA would first determine the relative concentrations of the pure magnesium and pure aluminum initially loaded into the smelting furnace in order to determine its solidus temperature. As a result, a POSITA would have understood in April 2014 that Example 7 of Xiao includes 62.85 total grams of magnesium.</p> <p>The following intermediate alloy compositions were chosen for the initial mass balance calculations: Al-Fe (30 wt% Fe), Al-Ni (45 wt% Ni), Al-Cu (40 wt% Cu), Al-Ti (10 wt% Ti), Al-Zr (20 wt% Zr) alloy mixtures. A POSITA in April 2014 would have performed the initial mass balances, included herewith as the Mass/Balance Exhibit 2, on these compositions. Using these initially selected alloy compositions would have resulted in 7.79 grams of Aluminum being added as components of the intermediate alloys. Since Example 7 of Xiao calls for a total of 25 grams (i.e. 25 wt%) of Aluminum, 17.21 grams of</p>
--	--

	<p>Aluminum would have had to have been initially loaded into the smelting furnace in Example 7 along with the 62.85 grams of magnesium (see calculation above). Thus, the initial melt of Example 7 (using the intermediate alloys selected) would have had a composition of 78.5 wt% Mg and 21.5 wt% Al.</p> <p>A POSITA in April 2014 would have next consulted the Mg-Al phase diagram to determine that the solidus temperature of that initial magnesium alloy (i.e. 78.5 wt% Mg and 21.5 wt% Al) would be 437 °C. A POSITA would also consult the relevant phase diagram to determine the melting point temperature of the various additive materials disclosed in Xiao. Id. at ¶ 122. An aluminum-iron alloy with 30 wt% iron would have a melting point temperature of 655 °C, an aluminum-nickel alloy with 45 wt% Ni would have a melting point temperature of 854 °C, an aluminum-copper alloy with 40 wt% Cu would have a melting point temperature of 548°C, an aluminum-titanium alloy with 10 wt% Ti would have a melting point temperature of 665°C, and an aluminum-zirconium alloy with 20 wt% Zr would have a melting point temperature of 661°C. Thus, each of these additive materials from Example 7 of Xiao (with the constituencies selected above) would have had a greater melting point temperature than the solidus temperature (i.e. 437°C) of the magnesium-aluminum alloy (78.5 wt% Mg and 21.5 wt% Al) calculated as the amount of pure aluminum and magnesium initially loaded into the smelting furnace in Example 7.</p> <p>A POSITA in April 2014 would have recognized that other compositions would have fallen within the teachings of Example 7 and other disclosures of Xiao resulting in Al-Fe, Al-Ni, and Al-Cu intermediate alloys having greater melting point temperatures than the solidus temperature of initial magnesium</p>
--	---

	alloy loaded in the various disclosures of Xiao. Thus, Xiao discloses all of the limitations of this claim element.
said additive material constituting about 0.05 wt %-45 wt % of said mixture,	<p>Claim 1 of Xiao discloses: a “magnesium alloy, comprising the components at the weight percentages as follows: Al: 13 to 25%, Zn: 2 to 15%, the remainder is Mg, and a sum of the weight percentages of the components is 100%.” Claim 2 discloses: a “magnesium alloy, according to Claim 1, further comprising the trace elements at the weight percentages as follows: Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%; and a sum of the weight percentages of the components is 100%.”</p> <p>These metallic elements are found in the additive materials generally disclosed by Xiao (i.e., intermediate alloys: Al-Fe, Al-Ni, Al-Cu, Al-Ti) (¶ 0023) and particularly described in association with Example 7 of Xiao (Id. at ¶¶ 0057–0060) for which a POSITA could calculate the specific weight percentage (ranges) of each additive material or secondary metal. In doing so, a POSITA would determine that the additive materials aluminum-iron with 30 wt% iron, aluminum-nickel with 45 wt% nickel, and aluminum-copper with 40 wt% copper also fall well within the range of about 0.05 wt% to 45 wt% of the mixture. For example, the aluminum-iron additive constitutes 3.3 wt% of the mixture; the aluminum-nickel additive constitutes 1.1 wt% of the mixture; and the aluminum-copper additive constitutes 0.25 wt% of the mixture. A POSITA in April 2014 would have also recognized that other intermediate alloy compositions would have fallen within the teachings of Example 7 and other disclosures of Xiao resulting in Al-Fe, Al-Ni, and Al-Cu intermediate alloys constituting about 0.05 wt%</p>

	<p>to 45 wt% of the mixture. Accordingly, this claim element was disclosed by Xiao.</p>
said additive material forming precipitant in said magnesium composite,	<p>Xiao teaches said additive material forming precipitant in said magnesium composite. Specifically, Xiao discloses a novel magnesium alloy with high aluminum and zinc content further adding elements Fe, Cu, Ni and Ag which can form intermetallics and enhance the corrosion performance of the magnesium alloy. ¶ 0026. Xiao explains the corrosion mechanism within a magnesium alloy in terms of a cathode phase, which Xiao explains includes “the <math>\beta</math> (Mg17Al12) phase and eutectic phase on the grain boundary of the magnesium alloy,” and “the magnesium matrix <math>\alpha</math> phase which functions as an anode phase, so that the matrix and the grain boundary of the magnesium alloy form a large amount of micro-batteries, which [micro- batteries] greatly accelerate the corrosion decomposition of magnesium alloy.” Id.</p> <p>A POSITA in August 2014 would have understood this disclosure of “intermetallic composite micro-particles” to be a reference to intermetallic precipitate. Medlin Decl. Exhibit 2 at ¶ 107.</p> <p>A POSITA reading the Xiao reference in April 2014 would have recognized that the precipitation of galvanically-active intermetallic phases (“micro-batteries”) are naturally and necessarily generated by the in situ reaction between magnesium and any and all of the disclosed metal additives forming in situ precipitate. Medlin Decl. Exhibit 2, at ¶ 108. Thus, these claim elements were disclosed by Xiao.</p>

	These elements are among the claimed “secondary metals” disclosed by Xiao. Thus, all of the limitations of this claim element are disclosed by Xiao.
said additive material includes one or more metals selected from the group consisting of copper, nickel, iron, and cobalt,	Xiao teaches the use of a variety of metals as additive materials. See, e.g., ¶ 0026. For instance, Xiao claims that the novel magnesium alloy may “further compris[e] trace elements at the weight percentages as follows: Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%. . .” Id. at Claim 2. In addition, Example 7 of Xiao particularly discloses intermediate alloys Al-Fe, Al-Ni, Al-Ag, Al-Cu, Al-Zr, Al-Ti and Zinc as additive materials. Id. at ¶ 0060. A POSITA in April 2014 would have understood that all of these intermediate alloys contain metal, including, in particular, copper, nickel, and iron, among other metals. Thus, Xiao discloses a number of metal additives, including copper, nickel, and iron and, hence fully discloses all of the limitations of these claim elements.
said magnesium composite has a dissolution rate of at least 5 mg/cm <sup>2</sup> /hr. in 3 wt % KCl water mixture at 90°C.	A POSITA in April 2014 would have recognized that Xiao disclosed a table of dissolution rates (“decomposition rates”) because the units of measurement are the units used by POSITAs to measure dissolution rates. The disclosed table provides seven examples (excluding the comparative [prior art] example). ¶ 0064.  Xiao provides dissolution rates in g/cm <sup>2</sup> /hr, while the claims of the ’653 Patent express dissolution rates in mg/cm <sup>2</sup> /hr. A POSITA would have known converting between g/cm <sup>2</sup> /hr to mg/cm <sup>2</sup> /hr, merely requires multiplication by 1,000.  Thus, each of Xiao’s seven examples (excluding the comparative [prior art] example) has a dissolution rate of at least three times the claimed “at least 5 mg/cm <sup>2</sup> /hr in 3 wt.% KCl water” at both 70°C or 93°C (with Example 7 being more than

	seven times that floor). A POSITA in April 2014 would have recognized that the dissolution rate of the examples in Xiao would necessarily have been well above the claimed floor of “at least of 5 mg/cm <sup>2</sup> /hr” at the claimed 90°C.
<b>Dependent Claim 2</b>	<b>Disclosure of Claim Element in Xiao</b>
2. The magnesium composite as defined in claim 1, wherein said magnesium alloy includes over 50 wt % magnesium and one or more metals selected from the group consisting of aluminum, boron, bismuth, zinc, zirconium, and manganese.	Xiao discloses a “magnesium alloy...and further adds elements of Fe, Cu, Ni, and Ag which can enhance the corrosion performance of the magnesium alloy...” ¶ 0026, ll. 1-3.  Claim 1 of Xiao specifically discloses a “magnesium alloy, comprising the components at the weight percentages as follows: Al: 13 to 25%, Zn: 2 to 15%; the remainder is Mg, and a sum of the weight percentages of the components is 100%.”  Claim 1 (emphasis added). Thus, Xiao discloses a range of magnesium 54.5-85 wt%, which overlaps and covers much of the claimed range set forth in these claim elements.  Xiao teaches 0.05-0.5 wt% zirconium and 2-15 wt% zinc. A claim is invalid as anticipated when it is in Markush form and a prior art reference discloses one of the claimed elements. See Fresenius USA, Inc. v. Baxter Int'l, Inc., 582 F.3d 1288, 1298 (Fed. Cir. 2009). See also Abbott Labs. v. Baxter Pharm. Prods., Inc., 334 F.3d 1274, 1280 (Fed.Cir.2003); Schering Corp. v. Geneva Pharms., Inc., 339 F.3d 1373, 1380 (Fed.Cir.2003); Thus, Xiao teaches all of these claim limitations.
<b>Dependent Claim 3</b>	<b>Disclosure of Claim Element in Xiao</b>
3. The magnesium composite as defined in claim 1, wherein said magnesium alloy includes over 50 wt % magnesium and one or more metals selected from the	Xiao discloses a “magnesium alloy...and further adds elements of Fe, Cu, Ni, and Ag which can enhance the corrosion performance of the magnesium alloy...” ¶ 0026, ll. 1-3.  Claim 1 of Xiao discloses a “magnesium alloy, comprising the components at the weight percentages as follows: Al: 13 to

<p>group consisting of aluminum in an amount of about 0.5-10 wt %, zinc in amount of about 0.1-3 wt %, zirconium in an amount of about 0.01-1 wt %, manganese in an amount of about 0.15-2 wt %, boron in amount of about 0.0002-0.04 wt %, and bismuth in amount of about 0.4-0.7 wt %.</p>	<p>25%, Zn: 2 to 15%; the remainder is Mg, and a sum of the weight percentages of the components is 100%.” Claim 1. Thus, Xiao discloses a range of magnesium 54.5-85 wt%, which overlaps and covers much of the claimed range set forth in these claim elements.</p> <p>This claim element is taught solely by the zirconium concentrations falling within the 0.01-3 wt% range. Claim 3 also requires, in addition to a similar zirconium concentration to the Markush claims, a zinc concentration of 0.1-3 wt%. Xiao teaches 0.05-0.5 wt% zirconium, which touches and covers at least a portion of these claimed ranges. Xiao similarly teaches 2-15 wt% zinc, which covers much of the claimed (0.1-3 wt%) zinc concentration. A claim is invalid as anticipated when it is in Markush form and a prior art reference disclosed one of the claimed elements. See Fresenius USA, Inc. v. Baxter Int'l, Inc., 582 F.3d 1288, 1298 (Fed. Cir. 2009). See also Abbott Labs. v. Baxter Pharm. Prods., Inc., 334 F.3d 1274, 1280 (Fed.Cir.2003); Schering Corp. v. Geneva Pharms., Inc., 339 F.3d 1373, 1380 (Fed.Cir.2003); Thus, Xiao teaches all of these claim limitations.</p>
Dependent Claim 4	Disclosure of Claim Element in Xiao
<p>4. The magnesium composite as defined in claim 1, wherein said additive material includes nickel,</p>	<p>Xiao discloses a “magnesium alloy...and further adds elements of Fe, Cu, Ni, and Ag which can enhance the corrosion performance of the magnesium alloy...” ¶ 0026, ll. 1-3. Xiao teaches the use of a variety of metals as additive materials. See, e.g., ¶ 0026. For instance, Xiao claims that the novel magnesium alloy may “further compris[e] trace elements at the weight percentages as follows: Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%...” Id. at Claim 2. In addition, Example 7 of Xiao particularly discloses intermediate alloys Al-Fe, Al-Ni, Al-Ag, Al-Cu, Al-Zr, Al-Ti</p>

	and Zinc as additive materials. Id. at ¶ 0060. A POSITA in April 2014 would have understood that all of these intermediate alloys contain metal, including, in particular, nickel, among other metals. Thus, Xiao discloses a number of metal additives, including nickel, and hence fully discloses all of the limitations of this claim element.
said nickel constitutes about 0.05-35 wt % of said magnesium composite,	<p>Claim 1 of Xiao discloses: a “magnesium alloy, comprising the components at the weight percentages as follows: Al: 13 to 25%, Zn: 2 to 15%, the remainder is Mg, and a sum of the weight percentages of the components is 100%.” Claim 2 discloses: a “magnesium alloy, according to Claim 1, further comprising the trace elements at the weight percentages as follows: Fe: 0.1 to 5%, Cu: 0.05 to 5%, <u>Ni: 0.05 to 5%</u>, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%; and a sum of the weight percentages of the components is 100%.” [Emphasis Added]</p> <p>These metallic elements are found in the additive materials generally disclosed by Xiao (i.e., intermediate alloys: Al-Fe, Al-Ni, Al-Cu, Al-Ti) (¶ 0023) and particularly described in association with Example 7 of Xiao (Id. at ¶¶ 0057–0060) for which a POSITA could calculate the specific weight percentage (ranges) of each additive material or secondary metal. In doing so, a POSITA would determine that the additive materials aluminum-iron with 30 wt% iron, aluminum-nickel with 45 wt% nickel, and aluminum-copper with 40 wt% copper also fall well within the range of about 0.05 wt% to 45 wt% of the mixture. For example, the aluminum-iron additive constitutes 3.3 wt% of the mixture; the aluminum-nickel additive constitutes 1.1 wt% of the mixture; and the aluminum-copper additive constitutes 0.25 wt% of the mixture. A POSITA in April 2014 would have also recognized that other intermediate alloy compositions would have fallen within the teachings of</p>

	Example 7 and other disclosures of Xiao resulting in Al-Fe, Al-Ni, and Al-Cu intermediate alloys constituting about 0.05 wt% to 35 wt% of the mixture. Accordingly, this claim element was disclosed by Xiao.
<b>Dependent Claim 5</b>	<b>Disclosure of Claim Element in Xiao</b>
5. The magnesium composite as defined in claim 1, wherein said additive material includes copper,	Xiao discloses a “magnesium alloy...and further adds elements of Fe, Cu, Ni, and Ag which can enhance the corrosion performance of the magnesium alloy...” ¶ 0026, ll. 1-3. Xiao teaches the use of a variety of metals as additive materials. See, e.g., ¶ 0026. For instance, Xiao claims that the novel magnesium alloy may “further compris[e] trace elements at the weight percentages as follows: Fe: 0.1 to 5%, <u>Cu: 0.05 to 5%</u> , Ni: 0.05 to 5%, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%...” Id. at Claim 2. [Emphasis Added]. In addition, Example 7 of Xiao particularly discloses intermediate alloys Al-Fe, Al-Ni, Al-Ag, Al-Cu, Al-Zr, Al-Ti and Zinc as additive materials. Id. at ¶ 0060. A POSITA in April 2014 would have understood that all of these intermediate alloys contain metal, including, in particular, copper, among other metals. Thus, Xiao discloses a number of metal additives, including copper, and hence fully discloses all of the limitations of these claim elements.
said copper constitutes about 0.05-35 wt % of said magnesium composite,	Claim 1 of Xiao discloses: a “magnesium alloy, comprising the components at the weight percentages as follows: Al: 13 to 25%, Zn: 2 to 15%, the remainder is Mg, and a sum of the weight percentages of the components is 100%.” Claim 2 discloses: a “magnesium alloy, according to Claim 1, further comprising the trace elements at the weight percentages as follows: Fe: 0.1 to 5%, <u>Cu: 0.05 to 5%</u> , Ni: 0.05 to 5%, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%; and a sum of the weight percentages of the components is 100%.” [Emphasis Added]

	<p>These metallic elements are found in the additive materials generally disclosed by Xiao (i.e., intermediate alloys: Al-Fe, Al-Ni, Al-Cu, Al-Ti) (¶ 0023) and particularly described in association with Example 7 of Xiao (Id. at ¶¶ 0057–0060) for which a POSITA could calculate the specific weight percentage (ranges) of each additive material or secondary metal. In doing so, a POSITA would determine that the additive materials aluminum-iron with 30 wt% iron, aluminum-nickel with 45 wt% nickel, and aluminum-copper with 40 wt% copper also fall well within the range of about 0.05 wt% to 45 wt% of the mixture. For example, the aluminum-iron additive constitutes 3.3 wt% of the mixture; the aluminum-nickel additive constitutes 1.1 wt% of the mixture; and the aluminum-copper additive constitutes 0.25 wt% of the mixture. A POSITA in April 2014 would have also recognized that other intermediate alloy compositions would have fallen within the teachings of Example 7 and other disclosures of Xiao resulting in Al-Fe, Al-Ni, and Al-Cu intermediate alloys constituting about 0.05 wt% to 35 wt% of the mixture. Accordingly, this claim element was disclosed by Xiao.</p>
said copper forms galvanically-active in situ precipitate in said magnesium composite.	Xiao discloses a novel magnesium alloy with high aluminum and zinc content further adding elements Fe, Cu, Ni and Ag which can form intermetallics and enhance the corrosion performance of the magnesium alloy. ¶ 0026. Xiao explains the corrosion mechanism within a magnesium alloy in terms of a cathode phase, which Xiao explains includes “the $\beta$ ( $Mg_{17}Al_{12}$ ) phase and eutectic phase on the grain boundary of the magnesium alloy,” and “the magnesium matrix $\alpha$ phase which functions as an anode phase, so that the matrix and the grain boundary of the magnesium alloy form a large amount of

	<p>micro-batteries, which [micro-batteries] greatly accelerate the corrosion decomposition of magnesium alloy.” Id.</p> <p>A POSITA in August 2014 would have understood this disclosure of “intermetallic composite micro-particles” to be a reference to intermetallic precipitate. Medlin Decl. Exhibit 2 at ¶ 107.</p> <p>A POSITA reading the Xiao reference in April 2014 would have recognized that the precipitation of galvanically-active intermetallic phases (“micro-batteries”) are naturally and necessarily generated by the <i>in situ</i> reaction between magnesium and any and all of the disclosed metal additives forming <i>in situ</i> precipitate. Medlin Decl. Exhibit 2, at ¶ 108. Thus, these claim elements were disclosed by Xiao. These elements are among the claimed “secondary metals” disclosed by Xiao. Thus, all of the limitations of this claim element are disclosed by Xiao.</p>
<b>Dependent Claim 9</b>	<b>Disclosure of Claim Element in Xiao</b>
9. The magnesium composite as defined in claim 1, wherein a dissolution rate of said magnesium composite is about 5-325 mg/cm <sup>2</sup> /hr. in 3 wt % KCl water mixture at 90°C.	<p>A POSITA in April 2014 would have recognized that Xiao disclosed a table of dissolution rates (“decomposition rates”) because the units of measurement are the units used by POSITAs to measure dissolution rates. The disclosed table provides seven examples (excluding the comparative [prior art] example). ¶ 0064.</p> <p>Xiao provides dissolution rates in g/cm<sup>2</sup>/hr, while the claims of the ’653 Patent express dissolution rates in mg/cm<sup>2</sup>/hr. A POSITA would have known converting between g/cm<sup>2</sup>/hr to mg/cm<sup>2</sup>/hr, merely requires multiplication by 1,000.</p>

	Thus, each of Xiao's seven examples (excluding the comparative [prior art] example) has a dissolution rate within the claimed range of "about 5-325 mg/cm <sup>2</sup> /hr in 3 wt.% KCl water" at both 70°C or 93°C (with Example 7 being more than seven times that floor). A POSITA in April 2014 would have recognized that the dissolution rate of the examples in Xiao would necessarily have been well above the claimed floor of "about 5" mg/cm <sup>2</sup> /hr at the claimed 90°C.
<b>Dependent Claim 11</b>	<b>Disclosure of Claim Element in Xiao</b>
11. The magnesium composite as defined in claim 1, wherein said additive material is a metal or metal alloy.	Xiao discloses a "magnesium alloy...and further adds elements of Fe, Cu, Ni, and Ag which can enhance the corrosion performance of the magnesium alloy..." ¶ 0026, ll. 1-3.  Xiao teaches the use of a variety of metals as additive materials. See, e.g., ¶ 0026. For instance, Xiao claims that the novel magnesium alloy may "further compris[e] trace elements at the weight percentages as follows: Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%...". Id. at Claim 2. In addition, Example 7 of Xiao particularly discloses intermediate alloys Al-Fe, Al-Ni, Al-Ag, Al-Cu, Al-Zr, Al-Ti and Zinc as additive materials. Id. at ¶ 0060. A POSITA in April 2014 would have understood that all of these intermediate alloys contain metal(s). Accordingly, Example 7 of Xiao discloses this claim element.
<b>Independent Claim 29</b>	<b>Disclosure of Claim Element in Xiao</b>
29. A dissolvable magnesium alloy composite for use in a ball or other tool component in a well drilling or completion operation,	Xiao discloses a "magnesium alloy...and further adds elements of Fe, Cu, Ni, and Ag which can enhance the corrosion performance of the magnesium alloy..." Xiao at ¶ 0026, ll. 1-9. See Declaration of Dr. Dana Medlin (Medlin Decl. Exhibit 2, e.g., at ¶¶ 104-105 (Claim 1)).

	Xiao discloses that the cast magnesium alloy “can be used as a tripping ball material for a multi-stage sliding sleeve staged-fracturing technique.” ¶ 0001. A POSITA would have understood that a tripping ball was a type of ball or other tool component in an oil well drilling operation. Medlin Decl. Exhibit 2 at ¶ 148.
said dissolvable magnesium alloy composite comprising 60-95 wt % magnesium; 0.01-1 wt % zirconium; and about 0.05-45 wt % of a secondary metal	<p>Claim 1 of Xiao discloses a “magnesium alloy, comprising the components at the weight percentages as follows: Al: 13 to 25%, Zn: 2 to 15%; the remainder is Mg, and a sum of the weight percentages of the components is 100%.” Xiao at Claim 1 (emphasis added). Dr. Medlin opines that a POSITA would have understood that the total range of the non-magnesium elements disclosed in Claim 1 of Xiao is 15 to 40%, leaving 60-85 wt% for magnesium. Medlin Decl. Exhibit 2 at ¶ 149. Dr. Medlin also notes that the “Examples of Xiao disclose a range of magnesium from 54.5 wt% - 79.2 wt%.” Id. Thus, Xiao discloses a range of magnesium 54.5-85 wt%, which overlaps and covers much of the claimed ranges set forth in these claim elements.</p> <p>The zirconium concentrations taught by Xiao fall within the claimed 0.01-1 wt% range.</p> <p>Claim 1 of Xiao discloses “Al: 13 to 25%, Zn: 2 to 15%,” and Claim 2 of Xiao discloses secondary metals of “Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%; and a sum of the weight percentages of the components is 100%.” Thus, Xiao teaches 0.05-0.5 wt% zirconium, which touches and covers at least a portion of these claimed ranges. Medlin Decl. Exhibit 2 ¶ 150. Xiao similarly teaches 2-15 wt% zinc, which covers much of the claimed (0.05-6 wt%) zinc concentration. Id. at ¶¶ 167-68. A claim is invalid as anticipated when it is in Markush form and a prior art reference discloses</p>

	<p>one of the claimed elements. See Fresenius USA, Inc. v. Baxter Int'l, Inc., 582 F.3d 1288, 1298 (Fed. Cir. 2009). See also Abbott Labs. v. Baxter Pharm. Prods., Inc., 334 F.3d 1274, 1280 (Fed.Cir.2003); Schering Corp. v. Geneva Pharms., Inc., 339 F.3d 1373, 1380 (Fed.Cir.2003);</p> <p>These metallic elements are found in the additive materials generally disclosed by Xiao (i.e., intermediate alloys: Al-Fe, Al-Ni, Al-Cu, Al-Ti) (Xiao at ¶ 0023) and particularly described in association with Example 7 of Xiao (Id. at ¶¶ 0057–0060) for which Dr. Medlin calculated for Example 7 of Xiao (Medlin Decl. Exhibit 2 at ¶¶ 113–122) the specific weight percentage (ranges) of each additive material or secondary metal. In particular, Dr. Medlin determined that the additive materials aluminum-iron with 30 wt% iron, aluminum- nickel with 45 wt% nickel, and aluminum-copper with 40 wt% copper also fall well within the range of about 0.05 wt% to 45 wt% of the mixture. Id. ¶ 122. For example, the aluminum-iron additive constitutes 3.3 wt% of the mixture; the aluminum-nickel additive constitutes 1.1 wt% of the mixture; and the aluminum-copper additive constitutes 0.25 wt% of the mixture. Dr. Medlin expressed his opinion that a POSITA in April 2014 would have also recognized that other intermediate alloy compositions would have fallen within the teachings of Example 7 and other disclosures of Xiao resulting in Al-Fe, Al-Ni, and Al-Cu intermediate alloys constituting about 0.05 wt% to 45 wt% of the mixture. Id. at ¶ 125. Accordingly, this claim element was disclosed by Xiao.</p>
to form a galvanically-active intermetallic particle that promotes corrosion of said	Xiao discloses a novel magnesium alloy with high aluminum and zinc content further adding elements Fe, Cu, Ni and Ag which can form intermetallics and enhance the corrosion performance of the magnesium alloy. Xiao at ¶ 0026. Xiao

dissolvable magnesium alloy composite,	explains the corrosion mechanism within a magnesium alloy in terms of a cathode phase, which Xiao explains includes “the $\beta$ ( $Mg_{17}Al_{12}$ ) phase and eutectic phase on the grain boundary of the magnesium alloy,” and “the magnesium matrix $\alpha$ phase which functions as an anode phase, so that the matrix and the grain boundary of the magnesium alloy form a large amount of micro-batteries, which [micro-batteries] greatly accelerate the corrosion decomposition of magnesium alloy.” Id. These elements are among the claimed “secondary metals” disclosed by Xiao. Thus, all of the limitations of this claim element are disclosed by Xiao.
said secondary metal including one or more metals selected from the group consisting of copper, nickel, cobalt, titanium and iron,	Xiao teaches the use of a variety of metals as additive materials. See, e.g., Xiao at ¶ 0026. For instance, Xiao claims that the novel magnesium alloy may “further compris[e] trace elements at the weight percentages as follows: Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%...” Id. at Claim 2. In addition, Example 7 of Xiao particularly discloses intermediate alloys Al-Fe, Al-Ni, Al-Ag, Al-Cu, Al-Zr, Al-Ti and Zinc as additive materials. Id. at ¶ 0060. A POSITA in April 2014 would have understood that all of these intermediate alloys contain metal, including, in particular, copper, nickel, and iron, among other metals. Medlin Decl. Exhibit 2 at ¶ 145. Thus, Xiao discloses a number of metal additives, including copper, nickel, and iron and, hence fully discloses all of the limitations of these claim elements.
said magnesium alloy composite has a dissolution rate of at least 5 mg/cm <sup>2</sup> /hr. in 3 wt % KCl water mixture at 90°C.	A POSITA in April 2014 would have recognized that Xiao disclosed a table of dissolution rates (“decomposition rates”) because the units of measurement are the units used by POSITAs to measure dissolution rates. Medlin Decl. Exhibit 2 at ¶ 129. The disclosed table provides seven examples (excluding the comparative [prior art] example). Xiao at ¶ 0064.

	<p>Xiao provides dissolution rates in g/cm<sup>2</sup>/hr, while the claims of the '653 Patent express dissolution rates in mg/cm<sup>2</sup>/hr. A POSITA would have known converting between g/cm<sup>2</sup>/hr to mg/cm<sup>2</sup>/hr, merely requires multiplication by 1,000. Medlin Decl. Exhibit 2 at ¶ 130.</p> <p>Thus, each of Xiao's seven examples (excluding the comparative [prior art] example) has a dissolution rate of at least three times the claimed "at least 5 mg/cm<sup>2</sup>/hr in 3 wt.% KCl water" at both 70°C or 93°C (with Example 7 being more than seven times that floor), Dr. Medlin opines that a POSITA in April 2014 would have recognized that the dissolution rate of the examples in Xiao would necessarily have been well above the claimed floor of "at least of 5 mg/cm<sup>2</sup>/hr" at the claimed 90°C. Id. at ¶ 132.</p>
<b>Dependent Claim 30</b>	<b>Disclosure of Claim Element in Xiao</b>
30. The dissolvable magnesium alloy composite as defined in claim 29, wherein a dissolution rate of said magnesium alloy composite is 5-325 mg/cm <sup>2</sup> /hr. in 3 wt % KCl water mixture at 90°C.	<p>A POSITA in April 2014 would have recognized that Xiao disclosed a table of dissolution rates ("decomposition rates") because the units of measurement are the units used by POSITAs to measure dissolution rates. Medlin Decl. Exhibit 2 at ¶ 129. The disclosed table provides seven examples (excluding the comparative [prior art] example). Xiao at ¶ 0064. Xiao provides dissolution rates in g/cm<sup>2</sup>/hr, while the claims of the '653 Patent express dissolution rates in mg/cm<sup>2</sup>/hr. A POSITA would have known converting between g/cm<sup>2</sup>/hr to mg/cm<sup>2</sup>/hr, merely requires multiplication by 1,000. Medlin Decl. Exhibit 2 at ¶ 130.</p> <p>Thus, each of Xiao's seven examples (excluding the comparative [prior art] example) has a dissolution rate within the claimed range of "about 5-325 mg/cm<sup>2</sup>/hr in 3 wt.% KCl water" at both 70°C or 93°C (with Example 7 being more than seven times that floor), Dr. Medlin opines that a POSITA in</p>

	April 2014 would have recognized that the dissolution rate of the examples in Xiao would necessarily have been well above the claimed floor of “about 5” mg/cm <sup>2</sup> /hr at the claimed 90°C. Id. at ¶ 132.
<b>Dependent Claim 31</b>	<b>Disclosure of Claim Element in Xiao</b>
31. The dissolvable magnesium composite as defined in claim 29, wherein said secondary metal includes nickel,	<p>Xiao discloses a “magnesium alloy...and further adds elements of Fe, Cu, Ni, and Ag which can enhance the corrosion performance of the magnesium alloy...” Xiao at ¶ 0026, ll. 1–9. See Declaration of Dr. Dana Medlin (Medlin Decl. Exhibit 2, e.g., at ¶ 104-105 (Claim 1)).</p> <p>Xiao teaches the use of a variety of metals as additive materials. See, e.g., Xiao at ¶ 0026. For instance, Xiao claims that the novel magnesium alloy may “further compris[e] trace elements at the weight percentages as follows: Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%...”Id. at Claim 2. In addition, Example 7 of Xiao particularly discloses intermediate alloys Al-Fe, Al-Ni, Al-Ag, Al-Cu, Al-Zr, Al-Ti and Zinc as additive materials. Id. at ¶ 0060. A POSITA in April 2014 would have understood that all of these intermediate alloys contain metal, including nickel, among other metals. Medlin Decl. Exhibit 2, at ¶ 145. Thus, Xiao discloses a number of metal additives, including nickel, and hence fully discloses all of the limitations of this claim element.</p>
said nickel forms galvanically-active in situ precipitate in said magnesium composite.	Xiao discloses a novel magnesium alloy with high aluminum and zinc content further adding elements Fe, Cu, Ni and Ag which can form intermetallics and enhance the corrosion performance of the magnesium alloy. Xiao at ¶ 0026. Xiao explains the corrosion mechanism within a magnesium alloy in terms of a cathode phase, which Xiao explains includes “the $\beta$ ( $Mg_{17}Al_{12}$ ) phase and eutectic phase on the grain boundary of the magnesium alloy,” and “the magnesium matrix $\alpha$ phase

	<p>which functions as an anode phase, so that the matrix and the grain boundary of the magnesium alloy form a large amount of micro-batteries, which [micro-batteries] greatly accelerate the corrosion decomposition of magnesium alloy.” Id.</p> <p>A POSITA in August 2014 would have understood this disclosure of “intermetallic composite micro-particles” to be a reference to intermetallic precipitate. Medlin Decl. Exhibit 2, at ¶ 107.</p> <p>A POSITA reading the Xiao reference in April 2014 would have recognized that the precipitation of galvanically-active intermetallic phases (“micro-batteries”) are naturally and necessarily generated by the <i>in situ</i> reaction between magnesium and any and all of the disclosed metal additives forming <i>in situ</i> precipitate. Medlin Decl. Exhibit 2, at ¶ 108. Thus, these claim elements were disclosed by Xiao.</p> <p>These elements are among the claimed “secondary metals” disclosed by Xiao. Thus, all of the limitations of this claim element are disclosed by Xiao.</p>
<b>Dependent Claim 32</b>	<b>Disclosure of Claim Element in Xiao</b>
32. The dissolvable magnesium alloy composite as defined in claim 29, wherein said additive material is a metal or metal alloy.	<p>Xiao discloses a “magnesium alloy...and further adds elements of Fe, Cu, Ni, and Ag which can enhance the corrosion performance of the magnesium alloy...” Xiao at ¶ 0026, ll. 1–9. See Declaration of Dr. Dana Medlin (Medlin Decl. Exhibit 2, e.g., at ¶¶ 104–105 (Claim 1)).</p> <p>Xiao teaches the use of a variety of metals as additive materials. See, e.g., Xiao at ¶ 0026. For instance, Xiao claims that the magnesium alloy may “further compris[e] trace elements at the weight percentages as follows: Fe: 0.1 to 5%, Cu: 0.05 to 5%,</p>

	Ni: 0.05 to 5%, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%. . .” Id. at Claim 2. In addition, Example 7 of Xiao particularly discloses intermediate alloys Al-Fe, Al-Ni, Al-Ag, Al-Cu, Al-Zr, Al-Ti and Zinc as additive materials. Id. at ¶ 0060. A POSITA in April 2014 would have understood that all of these additive materials contain metal(s). Medlin Decl. Exhibit 2, at ¶ 145. Accordingly, Example 7 of Xiao discloses this claim element in accordance with Patent Owner’s definition of “additive material.”
<b>Independent Claim 37</b>	<b>Disclosure of Claim Element in Xiao</b>
37. A dissolvable magnesium alloy composite for use in a ball or other tool component in a well drilling or completion operation,	Xiao discloses a “magnesium alloy...and further adds elements of Fe, Cu, Ni, and Ag which can enhance the corrosion performance of the magnesium alloy...” Xiao at ¶ 0026, ll. 1–9. See Declaration of Dr. Dana Medlin (Medlin Decl. Exhibit 2, e.g., at ¶ 104-105 (Claim 1)).  Xiao discloses that the cast magnesium alloy “can be used as a tripping ball material for a multi-stage sliding sleeve staged-fracturing technique.” ¶ 0001. A POSITA would have understood that a tripping ball was a type of ball or other tool component in an oil well drilling operation. Medlin Decl. Exhibit 2, at ¶ 148. Thus, these claim elements were disclosed by Xiao.
said dissolvable magnesium alloy composite comprising 60-95 wt % magnesium; 0.05-6 wt % zinc; 0.01-1 wt % zirconium;	Claim 1 of Xiao discloses a “magnesium alloy, comprising the components at the weight percentages as follows: Al: 13 to 25%, Zn: 2 to 15%; the remainder is Mg, and a sum of the weight percentages of the components is 100%.” Xiao at Claim 1 (emphasis added). Dr. Medlin opines that a POSITA would have understood that the total range of the non-magnesium elements disclosed in Claim 1 of Xiao is 15 to 40%, leaving 60-85 wt% for magnesium. Medlin Decl. Exhibit 2, at ¶ 149. Dr. Medlin also notes that the “Examples of Xiao disclose a range of magnesium from 54.5 wt% - 79.2 wt%.” Id. Thus, Xiao

	<p>discloses a range of magnesium 54.5-85 wt%, which overlaps and covers much of the claimed ranges set forth in these claim elements.</p> <p>Claim 2 discloses: a “magnesium alloy, according to Claim 1, further comprising the trace elements at the weight percentages as follows: Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%; and a sum of the weight percentages of the components is 100%.” Id. at Claim 2.</p> <p>These metallic elements are found in the additive materials generally disclosed by Xiao (i.e., intermediate alloys: Al-Fe, Al-Ni, Al-Cu, Al-Ti) (Xiao at ¶ 0023) and particularly described in association with Example 7 of Xiao (Id. at ¶¶ 0057–0060) for which Dr. Medlin calculated for Example 7 of Xiao (Medlin Decl. Exhibit 2, at ¶¶ 113–122) the specific weight percentage (ranges) of each additive material or secondary metal. In particular, Dr. Medlin determined that the additive materials aluminum-iron with 30 wt% iron, aluminum-nickel with 45 wt% nickel, and aluminum-copper with 40 wt% copper also fall well within the range of about 0.05 wt% to 45 wt% of the mixture. Id. ¶ 122. For example, the aluminum-iron additive constitutes 3.3 wt% of the mixture; the aluminum-nickel additive constitutes 1.1 wt% of the mixture; and the aluminum-copper additive constitutes 0.25 wt% of the mixture. Dr. Medlin expressed his opinion that a POSITA in April 2014 would have also recognized that other intermediate alloy compositions would have fallen within the teachings of Example 7 and other disclosures of Xiao resulting in Al-Fe, Al-Ni, and Al-Cu intermediate alloys constituting about 0.05 wt% to 45 wt% of the mixture. Id. at ¶ 125. Accordingly, these claim elements are disclosed by Xiao.</p>
--	---

<p>and about 0.05-45 wt % of a secondary metal to form a galvanically-active intermetallic particle that promotes corrosion of said dissolvable magnesium alloy composite,</p>	<p>Xiao discloses a novel magnesium alloy with high aluminum and zinc content further adding elements Fe, Cu, Ni and Ag which can form intermetallics and enhance the corrosion performance of the magnesium alloy. Xiao at ¶ 0026. Xiao explains the corrosion mechanism within a magnesium alloy in terms of a cathode phase, which Xiao explains includes “the <math>\beta</math> (<math>Mg_{17}Al_{12}</math>) phase and eutectic phase on the grain boundary of the magnesium alloy,” and “the magnesium matrix <math>\alpha</math> phase which functions as an anode phase, so that the matrix and the grain boundary of the magnesium alloy form a large amount of micro-batteries, which [micro-batteries] greatly accelerate the corrosion decomposition of magnesium alloy.” Id.</p> <p>A POSITA in August 2014 would have understood this disclosure of “intermetallic composite micro-particles” to be a reference to intermetallic precipitate. Medlin Decl. Exhibit 2, at ¶ 107.</p> <p>A POSITA reading the Xiao reference in April 2014 would have recognized that the precipitation of galvanically-active intermetallic phases (“micro-batteries”) are naturally and necessarily generated by the in situ reaction between magnesium and any and all of the disclosed metal additives forming in situ precipitate. Medlin Decl. Exhibit 2, at ¶ 108. Thus, these claim elements were disclosed by Xiao.</p> <p>These elements are among the claimed “secondary metals” disclosed by Xiao. Thus, all of the limitations of this claim element are disclosed by Xiao.</p>
<p>said secondary metal including one or more metals</p>	<p>Xiao teaches the use of a variety of metals as additive materials. See, e.g., Xiao at ¶ 0026. For instance, Xiao claims that the</p>

selected from the group consisting of copper, nickel, cobalt, titanium and iron,	novel magnesium alloy may “further compris[e] trace elements at the weight percentages as follows: Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%...” Id. at Claim 2. In addition, Example 7 of Xiao particularly discloses intermediate alloys Al-Fe, Al-Ni, Al-Ag, Al-Cu, Al-Zr, Al-Ti and Zinc as additive materials. Id. at ¶ 0060. A POSITA in April 2014 would have understood that all of these intermediate alloys contain metal, including, in particular, copper, nickel, and iron, among other metals. Medlin Decl. Exhibit 2, at ¶ 145. Thus, Xiao discloses a number of metal additives, including copper, nickel, and iron and, hence fully discloses all of the limitations of these claim elements.
said magnesium alloy composite has a dissolution rate of at least 5 mg/cm <sup>2</sup> /hr. in 3 wt % KCl water mixture at 90°C.	A POSITA in April 2014 would have recognized that Xiao disclosed a table of dissolution rates (“decomposition rates”) because the units of measurement are the units used by POSITAs to measure dissolution rates. Medlin Decl. Exhibit 2, at ¶ 129. The disclosed table provides seven examples (excluding the comparative [prior art] example). Xiao at ¶ 0064. Xiao provides dissolution rates in g/cm <sup>2</sup> /hr, while the claims of the ’653 Patent express dissolution rates in mg/cm <sup>2</sup> /hr. A POSITA would have known converting between g/cm <sup>2</sup> /hr to mg/cm <sup>2</sup> /hr, merely requires multiplication by 1,000. Medlin Decl. Exhibit 2, at ¶ 130.  Thus, each of Xiao’s seven examples (excluding the comparative [prior art] example) has a dissolution rate of at least three times the claimed “at least 5 mg/cm <sup>2</sup> /hr in 3 wt.% KCl water” at both 70°C or 93°C (with Example 7 being more than seven times that floor), Dr. Medlin opines that a POSITA in April 2014 would have recognized that the dissolution rate of the examples in Xiao would necessarily have been well above

	the claimed floor of “at least of 5 mg/cm <sup>2</sup> /hr” at the claimed 90°C. Id. at ¶ 132.
<b>Dependent Claim 38</b>	<b>Disclosure of Claim Element in Xiao</b>
38. The dissolvable magnesium alloy composite as defined in claim 37, wherein a dissolution rate of said magnesium alloy composite is 5-325 mg/cm <sup>2</sup> /hr. in 3 wt % KCl water mixture at 90°C.	<p>A POSITA in April 2014 would have recognized that Xiao disclosed a table of dissolution rates (“decomposition rates”) because the units of measurement are the units used by POSITAs to measure dissolution rates. Medlin Decl. Exhibit 2, at ¶ 129. The disclosed table provides seven examples (excluding the comparative [prior art] example). Xiao at ¶ 0064. Xiao provides dissolution rates in g/cm<sup>2</sup>/hr, while the claims of the ’653 Patent express dissolution rates in mg/cm<sup>2</sup>/hr. A POSITA would have known converting between g/cm<sup>2</sup>/hr to mg/cm<sup>2</sup>/hr, merely requires multiplication by 1,000. Medlin Decl. Exhibit 2, at ¶ 130.</p> <p>Thus, each of Xiao’s seven examples (excluding the comparative [prior art] example) has a dissolution rate within the claimed range of of at least three times the claimed “about 5-325 mg/cm<sup>2</sup>/hr in 3 wt.% KCl water” at both 70°C or 93°C (with Example 7 being more than seven times that floor), Dr. Medlin opines that a POSITA in April 2014 would have recognized that the dissolution rate of the examples in Xiao would necessarily have been well above the claimed floor of “about 5” “about 5-325 mg/cm<sup>2</sup>/hr” at the claimed 90°C. Id. at ¶ 132.</p>
<b>Dependent Claim 39</b>	<b>Disclosure of Claim Element in Xiao</b>
39. The dissolvable magnesium composite as defined in claim 37, wherein said secondary metal includes nickel,	Xiao discloses a “magnesium alloy...and further adds elements of Fe, Cu, Ni, and Ag which can enhance the corrosion performance of the magnesium alloy...” Xiao at ¶ 0026, ll. 1–9. See Declaration of Dr. Dana Medlin (Medlin Decl. Exhibit 2, e.g., at ¶ 104-105 (Claim 1)).

	Xiao teaches the use of a variety of metals as additive materials. See, e.g., Xiao at ¶ 0026. For instance, Xiao claims that the novel magnesium alloy may “further compris[e] trace elements at the weight percentages as follows: Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%...” Id. at Claim 2. In addition, Example 7 of Xiao particularly discloses intermediate alloys Al-Fe, Al-Ni, Al-Ag, Al-Cu, Al-Zr, Al-Ti and Zinc as additive materials. Id. at ¶ 0060. A POSITA in April 2014 would have understood that all of these intermediate alloys contain metal, including nickel, among other metals. Medlin Decl. Exhibit 2, at ¶ 145. Thus, Xiao discloses a number of metal additives, including nickel, and hence fully discloses all of the limitations of these claim elements.
said nickel forms galvanically-active in situ precipitate in said magnesium composite.	Xiao discloses a novel magnesium alloy with high aluminum and zinc content further adding elements Fe, Cu, Ni and Ag which can form intermetallics and enhance the corrosion performance of the magnesium alloy. Xiao at ¶ 0026. Xiao explains the corrosion mechanism within a magnesium alloy in terms of a cathode phase, which Xiao explains includes “the $\beta$ ( $Mg_{17}Al_{12}$ ) phase and eutectic phase on the grain boundary of the magnesium alloy,” and “the magnesium matrix $\alpha$ phase which functions as an anode phase, so that the matrix and the grain boundary of the magnesium alloy form a large amount of micro-batteries, which [micro-batteries] greatly accelerate the corrosion decomposition of magnesium alloy.” Id.  A POSITA in August 2014 would have understood this disclosure of “intermetallic composite micro-particles” to be a reference to intermetallic precipitate. Medlin Decl. Exhibit 2, at ¶ 107.

	<p>A POSITA reading the Xiao reference in April 2014 would have recognized that the precipitation of galvanically-active intermetallic phases (“micro-batteries”) are naturally and necessarily generated by the <i>in situ</i> reaction between magnesium and any and all of the disclosed metal additives forming <i>in situ</i> precipitate. Medlin Decl. Exhibit 2, at ¶ 108. Thus, these claim elements were disclosed by Xiao.</p> <p>These elements are among the claimed “secondary metals” disclosed by Xiao. Thus, all of the limitations of this claim element are disclosed by Xiao.</p>
<b>Dependent Claim 40</b>	<b>Disclosure of Claim Element in Xiao</b>
40. The dissolvable magnesium alloy composite as defined in claim 37, wherein said additive material is a metal or metal alloy.	<p>Xiao discloses a “magnesium alloy...and further adds elements of Fe, Cu, Ni, and Ag which can enhance the corrosion performance of the magnesium alloy...” Xiao at ¶ 0026, ll. 1–9. See Declaration of Dr. Dana Medlin (Medlin Decl. Exhibit 2, e.g., at ¶ 104-105 (Claim 1)).</p> <p>Xiao teaches the use of a variety of metals as additive materials. See, e.g., Xiao at ¶ 0026. For instance, Xiao claims that the magnesium alloy may “further compris[e] trace elements at the weight percentages as follows: Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%...” Id. at Claim 2. In addition, Example 7 of Xiao particularly discloses intermediate alloys Al-Fe, Al-Ni, Al-Ag, Al-Cu, Al-Zr, Al-Ti and Zinc as additive materials. Id. at ¶ 0060. A POSITA in April 2014 would have understood that all of these additive materials contain metal(s). Medlin Decl. Exhibit 2, at ¶ 145. Accordingly, Example 7 of Xiao discloses this claim element in accordance with Patent Owner’s definition of “additive material.”</p>
<b>Independent Claim 41</b>	<b>Disclosure of Claim Element in Xiao</b>

<p>41. A dissolvable magnesium alloy composite for use in a ball or other tool component in a well drilling or completion operation,</p>	<p>Xiao discloses a “magnesium alloy...and further adds elements of Fe, Cu, Ni, and Ag which can enhance the corrosion performance of the magnesium alloy...” Xiao at ¶ 0026, ll. 1–9. See Declaration of Dr. Dana Medlin (Medlin Decl. Exhibit 2, e.g., at ¶ 104-105 (Claim 1)).</p> <p>Xiao discloses that the cast magnesium alloy “can be used as a tripping ball material for a multi-stage sliding sleeve staged-fracturing technique.” ¶ 0001. A POSITA would have understood that a tripping ball was a type of ball or other tool component in an oil well drilling operation. Medlin Decl. Exhibit 2, at ¶ 148. Thus, these claim elements were disclosed by Xiao.</p>
<p>said dissolvable magnesium alloy composite comprising over 50 wt % magnesium; one or more metals selected from the group consisting of 0.5-10 wt % aluminum, 0.1-2 wt % zinc, 0.01-1 wt % zirconium, and 0.15-2 wt % manganese;</p>	<p>Claim 1 of Xiao discloses a “magnesium alloy, comprising the components at the weight percentages as follows: Al: 13 to 25%, Zn: 2 to 15%; the remainder is Mg, and a sum of the weight percentages of the components is 100%.” Xiao at Claim 1 (emphasis added). Dr. Medlin opines that a POSITA would have understood that the total range of the non-magnesium elements disclosed in Claim 1 of Xiao is 15 to 40%, leaving 60-85 wt% for magnesium. Medlin Decl. Exhibit 2, at ¶ 149. Dr. Medlin also notes that the “Examples of Xiao disclose a range of magnesium from 54.5 wt% - 79.2 wt%.” Id. Thus, Xiao discloses a range of magnesium 54.5-85 wt%, which overlaps and covers much of the claimed ranges set forth in these claim elements.</p> <p>This claim element would be anticipated solely by the zirconium concentrations falling within the 0.01-1 and/or 0.01-3 wt% ranges. Claim 41 also requires, in addition to a similar zirconium concentration to the Markush claims, a zinc concentration of 0.1-2 wt%.</p>

	<p>Claim 1 of Xiao discloses “Al: 13 to 25%, Zn: 2 to 15%,” and Claim 2 of Xiao discloses secondary metals of “Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%; and a sum of the weight percentages of the components is 100%.” Thus, Xiao teaches 0.05-0.5 wt% zirconium, which touches and covers at least a portion of these claimed ranges. Medlin Decl. Exhibit 2, ¶ 150. Xiao similarly teaches 2-15 wt% zinc, which covers much of the claimed (0.05-6 wt%) zinc concentration. Id. at ¶¶ 167-68. A claim is invalid as anticipated when it is in Markush form and a prior art reference disclosed one of the claimed elements. See Fresenius USA, Inc. v. Baxter Int'l, Inc., 582 F.3d 1288, 1298 (Fed. Cir. 2009). See also Abbott Labs. v. Baxter Pharm. Prods., Inc., 334 F.3d 1274, 1280 (Fed.Cir.2003); Schering Corp. v. Geneva Pharms., Inc., 339 F.3d 1373, 1380 (Fed.Cir.2003); Thus, Xiao teaches all of these claim limitations.</p> <p>Claim 2 discloses: a “magnesium alloy, according to Claim 1, further comprising the trace elements at the weight percentages as follows: Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%; and a sum of the weight percentages of the components is 100%.” Id. at Claim 2. These metallic elements are found in the additive materials generally disclosed by Xiao (i.e., intermediate alloys: Al-Fe, Al-Ni, Al-Cu, Al-Ti) (Xiao at ¶ 0023) and particularly described in association with Example 7 of Xiao (Id. at ¶¶ 0057–0060) for which Dr. Medlin calculated for Example 7 of Xiao (Medlin Decl. Exhibit 2, at ¶¶ 113–122) the specific weight percentage (ranges) of each additive material or secondary metal. In particular, Dr. Medlin determined that the additive materials aluminum-iron with 30 wt% iron, aluminum-nickel with 45 wt% nickel, and aluminum-copper with 40 wt% copper also fall</p>
--	--

	<p>well within the range of about 0.05 wt% to 45 wt% of the mixture. Id. ¶ 122. For example, the aluminum-iron additive constitutes 3.3 wt% of the mixture; the aluminum-nickel additive constitutes 1.1 wt% of the mixture; and the aluminum-copper additive constitutes 0.25 wt% of the mixture. Dr. Medlin expressed his opinion that a POSITA in April 2014 would have also recognized that other intermediate alloy compositions would have fallen within the teachings of Example 7 and other disclosures of Xiao resulting in Al-Fe, Al-Ni, and Al-Cu intermediate alloys constituting about 0.05 wt% to 45 wt% of the mixture. Id. at ¶ 125. Accordingly, these claim elements are disclosed by Xiao.</p> <p>Xiao discloses a novel magnesium alloy with high aluminum and zinc content further adding elements Fe, Cu, Ni and Ag which can form intermetallics and enhance the corrosion performance of the magnesium alloy. Xiao at ¶ 0026. Xiao explains the corrosion mechanism within a magnesium alloy in terms of a cathode phase, which Xiao explains includes “the <math>\beta</math> (<math>Mg_{17}Al_{12}</math>) phase and eutectic phase on the grain boundary of the magnesium alloy,” and “the magnesium matrix <math>\alpha</math> phase which functions as an anode phase, so that the matrix and the grain boundary of the magnesium alloy form a large amount of micro-batteries, which [micro- batteries] greatly accelerate the corrosion decomposition of magnesium alloy.” Id.</p> <p>These elements are among the claimed “secondary metals” disclosed by Xiao. Thus, all of the limitations of this claim element are disclosed by Xiao.</p>
and about 0.05-45 wt % of a secondary metal to form a galvanically-active intermetallic particle that	A POSITA in August 2014 would have understood the disclosure in Xiao of “intermetallic composite micro-particles” to be a reference to intermetallic precipitate. Medlin Decl. Exhibit 2, at ¶ 107.

promotes corrosion of said dissolvable magnesium alloy composite,	A POSITA reading the Xiao reference in April 2014 would have recognized that the precipitation of galvanically-active intermetallic phases (“micro-batteries”) are naturally and necessarily generated by the <i>in situ</i> reaction between magnesium and any and all of the disclosed metal additives forming <i>in situ</i> precipitate. Medlin Decl. Exhibit 2, at ¶ 108. Thus, these claim elements were disclosed by Xiao.
said secondary metal including one or more metals selected from the group consisting of copper, nickel and cobalt,	Xiao teaches the use of a variety of metals as additive materials. See, e.g., Xiao at ¶ 0026. For instance, Xiao claims that the novel magnesium alloy may “further compris[e] trace elements at the weight percentages as follows: Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%...”Id. at Claim 2. In addition, Example 7 of Xiao particularly discloses intermediate alloys Al-Fe, Al-Ni, Al-Ag, Al-Cu, Al-Zr, Al-Ti and Zinc as additive materials. Id. at ¶ 0060. A POSITA in April 2014 would have understood that all of these intermediate alloys contain metal, including, in particular, copper, nickel, and iron, among other metals. Medlin Decl. Exhibit 2, at ¶ 145. Thus, Xiao discloses a number of metal additives, including copper, nickel, and iron and, hence fully discloses all of the limitations of these claim elements.
said magnesium alloy composite has a dissolution rate of at least 5 mg/cm <sup>2</sup> /hr. in 3 wt % KCl water mixture at 90°C.	A POSITA in April 2014 would have recognized that Xiao disclosed a table of dissolution rates (“decomposition rates”) because the units of measurement are the units used by POSITAs to measure dissolution rates. Medlin Decl. Exhibit 2, at ¶ 129. The disclosed table provides seven examples (excluding the comparative [prior art] example). Xiao at ¶ 0064. Xiao provides dissolution rates in g/cm <sup>2</sup> /hr, while the claims of the ’653 Patent express dissolution rates in mg/cm <sup>2</sup> /hr. A POSITA would have known converting between g/cm <sup>2</sup> /hr to

	<p>mg/cm<sup>2</sup>/hr, merely requires multiplication by 1,000. Medlin Decl. Exhibit 2, at ¶ 130.</p> <p>Thus, each of Xiao's seven examples (excluding the comparative [prior art] example) has a dissolution rate of at least three times the claimed "at least 5 mg/cm<sup>2</sup>/hr in 3 wt.% KCl water" at both 70°C or 93°C (with Example 7 being more than seven times that floor), Dr. Medlin opines that a POSITA in April 2014 would have recognized that the dissolution rate of the examples in Xiao would necessarily have been well above the claimed floor of "at least of 5 mg/cm<sup>2</sup>/hr" at the claimed 90°C. Id. at ¶ 132.</p>
<b>Dependent Claim 42</b>	<b>Disclosure of Claim Element in Xiao</b>
42. The dissolvable magnesium alloy composite as defined in claim 41, wherein a dissolution rate of said magnesium alloy composite is 5-325 mg/cm <sup>2</sup> /hr. in 3 wt % KCl water mixture at 90°C.	<p>A POSITA in April 2014 would have recognized that Xiao disclosed a table of dissolution rates ("decomposition rates") because the units of measurement are the units used by POSITAs to measure dissolution rates. Medlin Decl. Exhibit 2, at ¶ 129. The disclosed table provides seven examples (excluding the comparative [prior art] example). Xiao at ¶ 0064. Xiao provides dissolution rates in g/cm<sup>2</sup>/hr, while the claims of the '653 Patent express dissolution rates in mg/cm<sup>2</sup>/hr. A POSITA would have known converting between g/cm<sup>2</sup>/hr to mg/cm<sup>2</sup>/hr, merely requires multiplication by 1,000. Medlin Decl. Exhibit 2, at ¶ 130.</p> <p>Thus, each of Xiao's seven examples (excluding the comparative [prior art] example) has a dissolution rate within the claimed range of of at least three times the claimed "about 5-325 mg/cm<sup>2</sup>/hr in 3 wt.% KCl water" at both 70°C or 93°C (with Example 7 being more than seven times that floor), Dr. Medlin opines that a POSITA in April 2014 would have recognized that the dissolution rate of the examples in Xiao would necessarily have been well above the claimed floor of</p>

	“about 5” “about 5-325 mg/cm <sup>2</sup> /hr” at the claimed 90°C. Id. at ¶ 132.
Dependent Claim 43	Disclosure of Claim Element in Xiao
43. The dissolvable magnesium composite as defined in claim 41, wherein said secondary metal includes nickel,	<p>Xiao discloses a “magnesium alloy...and further adds elements of Fe, Cu, Ni, and Ag which can enhance the corrosion performance of the magnesium alloy...” Xiao at ¶ 0026, ll. 1–9. See Declaration of Dr. Dana Medlin (Medlin Decl. Exhibit 2, e.g., at ¶ 104-105 (Claim 1)).</p> <p>Xiao teaches the use of a variety of metals as additive materials. See, e.g., Xiao at ¶ 0026. For instance, Xiao claims that the novel magnesium alloy may “further compris[e] trace elements at the weight percentages as follows: Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%...”Id. at Claim 2. In addition, Example 7 of Xiao particularly discloses intermediate alloys Al-Fe, Al-Ni, Al-Ag, Al-Cu, Al-Zr, Al-Ti and Zinc as additive materials. Id. at ¶ 0060. A POSITA in April 2014 would have understood that all of these intermediate alloys contain metal, including, in particular, copper, nickel, and iron, among other metals. Medlin Decl. Exhibit 2, at ¶ 145. Thus, Xiao discloses a number of metal additives, including copper, nickel, and iron and, hence fully discloses all of the limitations of these claim elements.</p>
said nickel forms galvanically-active in situ precipitate in said magnesium composite.	Xiao discloses a novel magnesium alloy with high aluminum and zinc content further adding elements Fe, Cu, Ni and Ag which can form intermetallics and enhance the corrosion performance of the magnesium alloy. Xiao at ¶ 0026. Xiao explains the corrosion mechanism within a magnesium alloy in terms of a cathode phase, which Xiao explains includes “the $\beta$ ( $Mg_{17}Al_{12}$ ) phase and eutectic phase on the grain boundary of the magnesium alloy,” and “the magnesium matrix $\alpha$ phase which functions as an anode phase, so that the matrix and the

	<p>grain boundary of the magnesium alloy form a large amount of micro-batteries, which [micro-batteries] greatly accelerate the corrosion decomposition of magnesium alloy.” Id.</p> <p>A POSITA in August 2014 would have understood this disclosure of “intermetallic composite micro-particles” to be a reference to intermetallic precipitate. Medlin Decl. Exhibit 2, at ¶ 107.</p> <p>A POSITA reading the Xiao reference in April 2014 would have recognized that the precipitation of galvanically-active intermetallic phases (“micro-batteries”) are naturally and necessarily generated by the <i>in situ</i> reaction between magnesium and any and all of the disclosed metal additives forming <i>in situ</i> precipitate. Medlin Decl. Exhibit 2, at ¶ 108. Thus, these claim elements were disclosed by Xiao.</p> <p>These elements are among the claimed “secondary metals” disclosed by Xiao. Thus, all of the limitations of this claim element are disclosed by Xiao.</p>
Dependent Claim 44	Disclosure of Claim Element in Xiao
44. The dissolvable magnesium alloy composite as defined in claim 41, wherein said additive material is a metal or metal alloy.	<p>Xiao discloses a “magnesium alloy...and further adds elements of Fe, Cu, Ni, and Ag which can enhance the corrosion performance of the magnesium alloy...” Xiao at ¶ 0026, ll. 1–9. See Declaration of Dr. Dana Medlin (Medlin Decl. Exhibit 2, e.g., at ¶ 104-105 (Claim 1)).</p> <p>Xiao teaches the use of a variety of metals as additive materials. See, e.g., Xiao at ¶ 0026. For instance, Xiao claims that the magnesium alloy may “further compris[e] trace elements at the weight percentages as follows: Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%...” Id. at Claim 2. In addition, Example 7 of Xiao particularly discloses</p>

	intermediate alloys Al-Fe, Al-Ni, Al-Ag, Al-Cu, Al-Zr, Al-Ti and Zinc as additive materials. Id. at ¶ 0060. A POSITA in April 2014 would have understood that all of these additive materials contain metal(s). Medlin Decl. Exhibit 2, at ¶ 145. Accordingly, Example 7 of Xiao discloses this claim element in accordance with Patent Owner's definition of "additive material."
<b>Independent Claim 45</b>	<b>Disclosure of Claim Element in Xiao</b>
45. A dissolvable magnesium alloy composite for use in a ball or other tool component in a well drilling or completion operation,	<p>Xiao discloses a "magnesium alloy...and further adds elements of Fe, Cu, Ni, and Ag which can enhance the corrosion performance of the magnesium alloy..." Xiao at ¶ 0026, ll. 1–9. See Declaration of Dr. Dana Medlin (Medlin Decl. Exhibit 2, e.g., at ¶ 104-105 (Claim 1)).</p> <p>Xiao discloses that the cast magnesium alloy "can be used as a tripping ball material for a multi-stage sliding sleeve staged-fracturing technique." ¶ 0001. A POSITA would have understood that a tripping ball was a type of ball or other tool component in an oil well drilling operation. Medlin Decl. Exhibit 2 at ¶ 148. Thus, these claim elements were disclosed by Xiao.</p> <p>Claim 2 discloses: a "magnesium alloy, according to Claim 1, further comprising the trace elements at the weight percentages as follows: Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%; and a sum of the weight percentages of the components is 100%." Id. at Claim 2.</p> <p>These metallic elements are found in the additive materials generally disclosed by Xiao (i.e., intermediate alloys: Al-Fe, Al-Ni, Al-Cu, Al-Ti) (Xiao at ¶ 0023) and particularly described in association with Example 7 of Xiao (Id. at ¶¶ 0057–0060) for which Dr. Medlin calculated for Example 7 of Xiao (Medlin Decl. Exhibit 2 at ¶¶ 113–122) the specific weight percentage (ranges) of each additive material or secondary metal. In</p>

	<p>particular, Dr. Medlin determined that the additive materials aluminum-iron with 30 wt% iron, aluminum-nickel with 45 wt% nickel, and aluminum-copper with 40 wt% copper also fall well within the range of about 0.05 wt% to 45 wt% of the mixture. Id. ¶ 122. For example, the aluminum-iron additive constitutes 3.3 wt% of the mixture; the aluminum-nickel additive constitutes 1.1 wt% of the mixture; and the aluminum-copper additive constitutes 0.25 wt% of the mixture. Dr. Medlin expressed his opinion that a POSITA in April 2014 would have also recognized that other intermediate alloy compositions would have fallen within the teachings of Example 7 and other disclosures of Xiao resulting in Al-Fe, Al-Ni, and Al-Cu intermediate alloys constituting about 0.05 wt% to 45 wt% of the mixture. Id. at ¶ 125. Accordingly, these claim elements are disclosed by Xiao.</p>
said dissolvable magnesium alloy composite comprising over 50 wt % magnesium; one or more metals selected from the group consisting of 0.1-3 wt % zinc, 0.01-1 wt % zirconium, 0.05-1 wt % manganese, 0.0002-0.04 wt % boron, and 0.4-0.7 wt % bismuth; and about 0.05-45 wt % of a secondary metal to form a galvanically-active intermetallic particle that promotes corrosion of said dissolvable magnesium alloy composite	<p>Claim 1 of Xiao discloses a “magnesium alloy, comprising the components at the weight percentages as follows: Al: 13 to 25%, Zn: 2 to 15%; the remainder is Mg, and a sum of the weight percentages of the components is 100%.” Xiao at Claim 1 (emphasis added). Dr. Medlin opines that a POSITA would have understood that the total range of the non-magnesium elements disclosed in Claim 1 of Xiao is 15 to 40%, leaving 60-85 wt% for magnesium. Medlin Decl. Exhibit 2 at ¶ 149. Dr. Medlin also notes that the “Examples of Xiao disclose a range of magnesium from 54.5 wt% - 79.2 wt%.” Id. Thus, Xiao discloses a range of magnesium 54.5-85 wt%, which overlaps and covers much of the claimed ranges set forth in these claim elements.</p> <p>All of the foregoing Markush claim elements (i.e., Claims 25, 41, 45, and 55) and this claim element of Claim 29 would be anticipated solely by the zirconium concentrations falling</p>

	<p>within the 0.01-1 and/or 0.01-3 wt% ranges. Claim 37 also requires, in addition to a similar zirconium concentration to the Markush claims, a zinc concentration of 0.05-6 wt%.</p> <p>Claim 1 of Xiao discloses “Al: 13 to 25%, Zn: 2 to 15%,” and Claim 2 of Xiao discloses secondary metals of “Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%; and a sum of the weight percentages of the components is 100%.” Thus, Xiao teaches 0.05-0.5 wt% zirconium, which touches and covers at least a portion of these claimed ranges. Medlin Decl. Exhibit 2 ¶ 150. Xiao similarly teaches 2-15 wt% zinc, which covers much of the claimed (0.05-6 wt%) zinc concentration. Id. at ¶¶ 167-68. A claim is invalid as anticipated when it is in Markush form and a prior art reference disclosed one of the claimed elements. See Fresenius USA, Inc. v. Baxter Int'l, Inc., 582 F.3d 1288, 1298 (Fed. Cir. 2009). See also Abbott Labs. v. Baxter Pharm. Prods., Inc., 334 F.3d 1274, 1280 (Fed.Cir.2003); Schering Corp. v. Geneva Pharms., Inc., 339 F.3d 1373, 1380 (Fed.Cir.2003); Thus, Xiao teaches all of these claim limitations.</p> <p>Xiao discloses a novel magnesium alloy with high aluminum and zinc content further adding elements Fe, Cu, Ni and Ag which can form intermetallics and enhance the corrosion performance of the magnesium alloy. Xiao at ¶ 0026. Xiao explains the corrosion mechanism within a magnesium alloy in terms of a cathode phase, which Xiao explains includes “the <math>\beta</math> (<math>Mg_{17}Al_{12}</math>) phase and eutectic phase on the grain boundary of the magnesium alloy,” and “the magnesium matrix <math>\alpha</math> phase which functions as an anode phase, so that the matrix and the grain boundary of the magnesium alloy form a large amount of micro-batteries, which [micro- batteries] greatly accelerate the corrosion decomposition of magnesium alloy.” Id.</p>
--	---

	These elements are among the claimed “secondary metals” disclosed by Xiao. Thus, all of the limitations of this claim element are disclosed by Xiao.
said secondary metal including one or more metals selected from the group consisting of copper, nickel, and cobalt,	Xiao teaches the use of a variety of metals as additive materials. See, e.g., Xiao at ¶ 0026. For instance, Xiao claims that the novel magnesium alloy may “further compris[e] trace elements at the weight percentages as follows: Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%...” Id. at Claim 2. In addition, Example 7 of Xiao particularly discloses intermediate alloys Al-Fe, Al-Ni, Al-Ag, Al-Cu, Al-Zr, Al-Ti and Zinc as additive materials. Id. at ¶ 0060. A POSITA in April 2014 would have understood that all of these intermediate alloys contain metal, including, in particular, copper, nickel, and iron, among other metals. Medlin Decl. Exhibit 2 at ¶ 145. Thus, Xiao discloses a number of metal additives, including copper, nickel, and iron and, hence fully discloses all of the limitations of these claim elements.
said magnesium alloy composite has a dissolution rate of at least 5 mg/cm <sup>2</sup> /hr. in 3 wt % KCl water mixture at 90°C.	A POSITA in April 2014 would have recognized that Xiao disclosed a table of dissolution rates (“decomposition rates”) because the units of measurement are the units used by POSITAs to measure dissolution rates. Medlin Decl. Exhibit 2 at ¶ 129. The disclosed table provides seven examples (excluding the comparative [prior art] example). Xiao at ¶ 0064. Xiao provides dissolution rates in g/cm <sup>2</sup> /hr, while the claims of the ’653 Patent express dissolution rates in mg/cm <sup>2</sup> /hr. A POSITA would have known converting between g/cm <sup>2</sup> /hr to mg/cm <sup>2</sup> /hr, merely requires multiplication by 1,000. Medlin Decl. Exhibit 2 at ¶ 130.  Thus, each of Xiao’s seven examples (excluding the comparative [prior art] example) has a dissolution rate of at least three times the claimed “at least 5 mg/cm <sup>2</sup> /hr in 3 wt.% KCl

	water” at both 70°C or 93°C (with Example 7 being more than seven times that floor), Dr. Medlin opines that a POSITA in April 2014 would have recognized that the dissolution rate of the examples in Xiao would necessarily have been well above the claimed floor of “at least of 5 mg/cm2/hr” at the claimed 90°C. Id. at ¶ 132.
<b>Dependent Claim 46</b>	<b>Disclosure of Claim Element in Xiao</b>
46. The dissolvable magnesium alloy composite as defined in claim 45, wherein a dissolution rate of said magnesium alloy composite is 5-325 mg/cm2/hr. in 3 wt % KCl water mixture at 90°C.	<p>A POSITA in April 2014 would have recognized that Xiao disclosed a table of dissolution rates (“decomposition rates”) because the units of measurement are the units used by POSITAs to measure dissolution rates. Medlin Decl. Exhibit 2 at ¶ 129. The disclosed table provides seven examples (excluding the comparative [prior art] example). Xiao at ¶ 0064. Xiao provides dissolution rates in g/cm2/hr, while the claims of the ’653 Patent express dissolution rates in mg/cm2/hr. A POSITA would have known converting between g/cm2/hr to mg/cm2/hr, merely requires multiplication by 1,000. Medlin Decl. Exhibit 2 at ¶ 130.</p> <p>Thus, each of Xiao’s seven examples (excluding the comparative [prior art] example) has a dissolution rate within the claimed range of “about 5-325 mg/cm2/hr in 3 wt.% KCl water” at both 70°C or 93°C (with Example 7 being more than seven times that floor), Dr. Medlin opines that a POSITA in April 2014 would have recognized that the dissolution rate of the examples in Xiao would necessarily have been well above the claimed floor of “about 5” mg/cm2/hr at the claimed 90°C. Id. at ¶ 132.</p>
<b>Dependent Claim 45</b>	<b>Disclosure of Claim Element in Xiao</b>
47. The dissolvable magnesium composite as defined in claim 45, wherein	Xiao discloses a “magnesium alloy...and further adds elements of Fe, Cu, Ni, and Ag which can enhance the corrosion performance of the magnesium alloy...” Xiao at ¶ 0026, ll. 1–9.

said secondary metal includes nickel,	<p>See Declaration of Dr. Dana Medlin (Medlin Decl. Exhibit 2, e.g., at ¶ 104-105 (Claim 1)).</p> <p>Xiao teaches the use of a variety of metals as additive materials. See, e.g., Xiao at ¶ 0026. For instance, Xiao claims that the novel magnesium alloy may “further compris[e] trace elements at the weight percentages as follows: Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%...”Id. at Claim 2. In addition, Example 7 of Xiao particularly discloses intermediate alloys Al-Fe, Al-Ni, Al-Ag, Al-Cu, Al-Zr, Al-Ti and Zinc as additive materials. Id. at ¶ 0060. A POSITA in April 2014 would have understood that all of these intermediate alloys contain metal, including nickel, among other metals. Medlin Decl. Exhibit 2 at ¶ 145. Thus, Xiao discloses a number of metal additives, including nickel, and hence fully discloses all of the limitations of these claim elements.</p>
said nickel forms galvanically-active in situ precipitate in said magnesium composite.	<p>Xiao discloses a novel magnesium alloy with high aluminum and zinc content further adding elements Fe, Cu, Ni and Ag which can form intermetallics and enhance the corrosion performance of the magnesium alloy. Xiao at ¶ 0026. Xiao explains the corrosion mechanism within a magnesium alloy in terms of a cathode phase, which Xiao explains includes “the <math>\beta</math> (<math>Mg_{17}Al_{12}</math>) phase and eutectic phase on the grain boundary of the magnesium alloy,” and “the magnesium matrix <math>\alpha</math> phase which functions as an anode phase, so that the matrix and the grain boundary of the magnesium alloy form a large amount of micro-batteries, which [micro-batteries] greatly accelerate the corrosion decomposition of magnesium alloy.” Id.</p> <p>A POSITA in August 2014 would have understood this disclosure of “intermetallic composite micro-particles” to be a</p>

	<p>reference to intermetallic precipitate. Medlin Decl. Exhibit 2 at ¶ 107.</p> <p>A POSITA reading the Xiao reference in April 2014 would have recognized that the precipitation of galvanically-active intermetallic phases (“micro-batteries”) are naturally and necessarily generated by the in situ reaction between magnesium and any and all of the disclosed metal additives forming in situ precipitate. Medlin Decl. Exhibit 2, at ¶ 108. Thus, these claim elements were disclosed by Xiao. These elements are among the claimed “secondary metals” disclosed by Xiao. Thus, all of the limitations of this claim element are disclosed by Xiao.</p>
<b>Dependent Claim 48</b>	<b>Disclosure of Claim Element in Xiao</b>
48. The dissolvable magnesium alloy composite as defined in claim 45, wherein said additive material is a metal or metal alloy.	<p>Xiao discloses a “magnesium alloy...and further adds elements of Fe, Cu, Ni, and Ag which can enhance the corrosion performance of the magnesium alloy...” Xiao at ¶ 0026, ll. 1–9. See Declaration of Dr. Dana Medlin (Medlin Decl. Exhibit 2, e.g., at ¶ 104-105 (Claim 1)).</p> <p>Xiao teaches the use of a variety of metals as additive materials. See, e.g., Xiao at ¶ 0026. For instance, Xiao claims that the magnesium alloy may “further compris[e] trace elements at the weight percentages as follows: Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%...” Id. at Claim 2. In addition, Example 7 of Xiao particularly discloses intermediate alloys Al-Fe, Al-Ni, Al-Ag, Al-Cu, Al-Zr, Al-Ti and Zinc as additive materials. Id. at ¶ 0060. A POSITA in April 2014 would have understood that all of these additive materials contain metal(s). Medlin Decl. Exhibit 2 at ¶ 145. Accordingly, Example 7 of Xiao discloses this claim element in accordance with Patent Owner’s definition of “additive material.”</p>

<b>Independent Claim 49</b>	<b>Disclosure of Claim Element in Xiao</b>
<p>49. A magnesium composite that includes in situ precipitation of galvanically-active intermetallic phases to enable controlled dissolution of said magnesium composite,</p>	<p>Xiao discloses a “magnesium alloy...and further adds elements of Fe, Cu, Ni, and Ag which can enhance the corrosion performance of the magnesium alloy...” Xiao at ¶ 0026, ll. 1–9. See Declaration of Dr. Dana Medlin (Medlin Decl. Exhibit 2, e.g., at ¶ 104-105 (Claim 1)).</p> <p>Xiao discloses a novel magnesium alloy with high aluminum and zinc content further adding elements Fe, Cu, Ni and Ag which can form intermetallics and enhance the corrosion performance of the magnesium alloy. Xiao explains the corrosion mechanism within a magnesium alloy in terms of a cathode phase, which Xiao explains includes “the <math>\beta</math> (<math>Mg_{17}Al_{12}</math>) phase and eutectic phase on the grain boundary of the magnesium alloy,” and “the magnesium matrix <math>\alpha</math> phase which functions as an anode phase, so that the matrix and the grain boundary of the magnesium alloy form a large amount of micro-batteries, which [micro-batteries] greatly accelerate the corrosion decomposition of magnesium alloy.” Xiao at ¶ 0026. A POSITA in August 2014 would have understood this disclosure of “intermetallic composite micro-particles” to be a reference to intermetallic precipitate. Medlin Decl. Exhibit 2 at ¶ 107.</p> <p>A POSITA reading the Xiao reference in April 2014 would have recognized that the precipitation of galvanically-active intermetallic phases (“micro-batteries”) are naturally and necessarily generated by the in situ reaction between magnesium and any and all of the disclosed metal additives forming in situ precipitate. Medlin Decl. Exhibit 2, at ¶ 108. Thus, these claim elements were disclosed by Xiao.</p>

said magnesium composite comprising a mixture of magnesium or a magnesium alloy and an additive material,	<p>Xiao discloses “first loading pure magnesium and pure aluminum into a smelting furnace . . . next loading pure zinc and an [sic] intermediate alloys of trace element components into a resulting magnesium-aluminum alloy melt after melting.” Xiao, at ¶ 0022. Xiao discloses that “Al-Fe . . . Al-Ni . . . Al-Cu . . . Al-Ag . . . Al-Zr . . . and . . . Al-Ti intermediate alloy[s], are heated to dry and then added to the magnesium-aluminum alloy melt.” Id. at ¶ 0023. These intermediate alloys meet the Patent Owner’s definition of “additive material,” i.e., “a material that is added.” Medlin Decl. Exhibit 2, at ¶ 110. The addition of these intermediate alloys to the melted magnesium alloy necessarily results in a mixture of the additive material and magnesium alloy. Id. at ¶ 111.</p>
said additive material constituting about 0.05-45 wt % of said mixture,	<p>Claim 1 of Xiao discloses: a “magnesium alloy, comprising the components at the weight percentages as follows: Al: 13 to 25%, Zn: 2 to 15%, the remainder is Mg, and a sum of the weight percentages of the components is 100%.” Xiao at Claim 1. And Claim 2 discloses: a “magnesium alloy, according to Claim 1, further comprising the trace elements at the weight percentages as follows: Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%; and a sum of the weight percentages of the components is 100%.” Id. at Claim 2.</p> <p>These metallic elements are found in the additive materials generally disclosed by Xiao (i.e., intermediate alloys: Al-Fe, Al-Ni, Al-Cu, Al-Ti) (Xiao at ¶ 0023) and particularly described in association with Example 7 of Xiao (Id. at ¶¶ 0057–0060) for which Dr. Medlin calculated for Example 7 of Xiao (Medlin Decl. Exhibit 2 at ¶¶ 113–122) the specific weight percentage (ranges) of each additive material or secondary metal. In particular, Dr. Medlin determined that the additive materials</p>

	aluminum-iron with 30 wt% iron, aluminum-nickel with 45 wt% nickel, and aluminum-copper with 40 wt% copper also fall well within the range of about 0.05 wt% to 45 wt% of the mixture. Id. ¶ 122. For example, the aluminum-iron additive constitutes 3.3 wt% of the mixture; the aluminum-nickel additive constitutes 1.1 wt% of the mixture; and the aluminum-copper additive constitutes 0.25 wt% of the mixture. Dr. Medlin expressed his opinion that a POSITA in April 2014 would have also recognized that other intermediate alloy compositions would have fallen within the teachings of Example 7 and other disclosures of Xiao resulting in Al-Fe, Al-Ni, and Al-Cu intermediate alloys constituting about 0.05 wt% to 45 wt% of the mixture. Id. at ¶ 125. Accordingly, these claim elements are disclosed by Xiao.
said additive material includes one or more metals selected from the group consisting of copper, nickel, titanium, iron, and cobalt,	Xiao teaches the use of a variety of metals as additive materials. See, e.g., Xiao at ¶ 0026. For instance, Xiao claims that the novel magnesium alloy may “further compris[e] trace elements at the weight percentages as follows: Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%...” Id. at Claim 2. In addition, Example 7 of Xiao particularly discloses intermediate alloys Al-Fe, Al-Ni, Al-Ag, Al-Cu, Al-Zr, Al-Ti and Zinc as additive materials. Id. at ¶ 0060. A POSITA in April 2014 would have understood that all of these intermediate alloys contain metal, including, in particular, copper, nickel, and iron, among other metals. Medlin Decl. Exhibit 2 at ¶ 145. Thus, Xiao discloses a number of metal additives, including copper, nickel, and iron and, hence fully discloses all of the limitations of these claim elements.
said magnesium composite including in situ precipitation of galvanically-active	Xiao discloses a novel magnesium alloy with high aluminum and zinc content further adding elements Fe, Cu, Ni and Ag which can form intermetallics and enhance the corrosion

intermetallic phases that include said additive material,	<p>performance of the magnesium alloy. Xiao at ¶ 0026. Xiao explains the corrosion mechanism within a magnesium alloy in terms of a cathode phase, which Xiao explains includes “the <math>\beta</math> (<math>Mg_{17}Al_{12}</math>) phase and eutectic phase on the grain boundary of the magnesium alloy,” and “the magnesium matrix <math>\alpha</math> phase which functions as an anode phase, so that the matrix and the grain boundary of the magnesium alloy form a large amount of micro-batteries, which [micro- batteries] greatly accelerate the corrosion decomposition of magnesium alloy.” Id.</p> <p>As Dr. Medlin explains, a POSITA in August 2014 would have understood this disclosure of “intermetallic composite micro-particles” to be a reference to intermetallic precipitate. Medlin Decl. Exhibit 2 at ¶ 256. As noted above, at least Fe, Cu, and Ni are “additive materials.” Consequently, the in-situ precipitation of galvanically- active intermetallic phases will include one or more of these additive materials. Thus, Xiao discloses all of these claim limitations.</p>
said additive material located in sufficient quantities in said galvanically-active intermetallic phases so as to obtain a composition and morphology of said galvanically-active intermetallic phases such that a galvanic corrosion rate along said galvanically-active intermetallic phases causes said magnesium composite to have a dissolution rate of at least at	<p>A POSITA would understand that the galvanically-active intermetallic phases (“composite micro-particles”) have a morphology that enhances the corrosion dissolution rate of the alloy due to a larger number of smaller particles throughout the microstructure. Medlin Decl. Exhibit 2 at ¶ 229. A POSITA would also understand the relationship between “composite micro-particles” and morphology of the galvanically-active intermetallic phases. Id.</p> <p>With respect to the “sufficient quantities” limitation, other claim limitations in Claims 49 and 73, define the weight percentage for the additive material as 0.05-45 wt% and about 0.05 wt%, respectively, which limitations are met by the teaching of Xiao with respect to four metals. In particular, Xiao discloses that “[e]lements such as Fe, Cu, Ni, Ag, etc., in the</p>

least 5 mg/cm <sup>2</sup> /hr. in 3 wt % KCl water mixture at 90°C.	<p>magnesium alloy can form a large number of intermetallic composite micro-particles, which can improve the corrosion performance of the magnesium alloy, thereby promoting the decomposition of the magnesium alloy.” Id. ¶ 0026. And the magnesium alloy may “further compris[e] trace elements at the weight percentages as follows: Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%;...” Id. at Claim 2. As such, Xiao discloses four metals (i.e., Fe, Cu, Ni, and Ti) included within the list of “additive material” set forth in Claim Element M, supra. The claimed weight % quantities of the “additive material” of Claim Element I, supra, also overlap with the weight % ranges of Xiao’s “trace elements”. As such, Claim Element P of claims 49 and 73-- that the “additive materials” are located in “sufficient quantities”—is fully disclosed by Xiao’s Claim 2 disclosure of “trace elements at the weight percentages as follows: Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%;...” Id. As such, Xiao’s four metal “trace elements” are present in “sufficient quantities” to meet Claim Element P of claims 49 and 73.</p> <p>A POSITA in April 2014 would have recognized that Xiao disclosed a table of dissolution rates (“decomposition rates”) because the units of measurement are the units used by POSITAs to measure dissolution rates. Medlin Decl. Exhibit 2 at ¶ 129. The disclosed table provides seven examples (excluding the comparative [prior art] example). Xiao at ¶ 0064. Xiao provides dissolution rates in g/cm<sup>2</sup>/hr, while the claims of the ’653 Patent express dissolution rates in mg/cm<sup>2</sup>/hr. A POSITA would have known converting between g/cm<sup>2</sup>/hr to mg/cm<sup>2</sup>/hr, merely requires multiplication by 1,000. Medlin Decl. Exhibit 2 at ¶ 130.</p>
--	--

	Thus, each of Xiao's seven examples (excluding the comparative [prior art] example) has a dissolution rate of at least three times the claimed "at least 5 mg/cm <sup>2</sup> /hr in 3 wt.% KCl water" at both 70°C or 93°C (with Example 7 being more than seven times that floor), Dr. Medlin opines that a POSITA in April 2014 would have recognized that the dissolution rate of the examples in Xiao would necessarily have been well above the claimed floor of "at least of 5 mg/cm <sup>2</sup> /hr" at the claimed 90°C. Id. at ¶ 132.
<b>Dependent Claim 50</b>	<b>Disclosure of Claim Element in Xiao</b>
50. The magnesium composite as defined in claim 49, wherein said additive material includes one or more metals selected from the group consisting of copper, nickel, and cobalt.	Xiao teaches the use of a variety of metals as additive materials. See, e.g., Xiao at ¶ 0026. For instance, Xiao claims that the novel magnesium alloy may "further compris[e] trace elements at the weight percentages as follows: Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%..." Id. at Claim 2. In addition, Example 7 of Xiao particularly discloses intermediate alloys Al-Fe, Al-Ni, Al-Ag, Al-Cu, Al-Zr, Al-Ti and Zinc as additive materials. Id. at ¶ 0060. A POSITA in April 2014 would have understood that all of these intermediate alloys contain metal, including, in particular, copper and nickel, among other metals. Medlin Decl. Exhibit 2 at ¶ 145. Thus, Xiao discloses a number of metal additives, including copper and nickel, and hence fully discloses all of the limitations of these claim elements.
<b>Dependent Claim 52</b>	<b>Disclosure of Claim Element in Xiao</b>
52. The magnesium composite as defined in claim 49, wherein said magnesium alloy includes over 50 wt % magnesium, and one or more metals selected from the	Xiao discloses a "magnesium alloy...and further adds elements of Fe, Cu, Ni, and Ag which can enhance the corrosion performance of the magnesium alloy..." Xiao at ¶ 0026, ll. 1–9. See Declaration of Dr. Dana Medlin (Medlin Decl. Exhibit 2, e.g., at ¶ 104-105 (Claim 1)).

group consisting of aluminum, boron, bismuth, zinc, zirconium, and manganese.	Claim 1 of Xiao discloses a “magnesium alloy, comprising the components at the weight percentages as follows: Al: 13 to 25%, Zn: 2 to 15%; the remainder is Mg, and a sum of the weight percentages of the components is 100%.” Xiao at Claim 1 (emphasis added). Dr. Medlin opines that a POSITA would have understood that the total range of the non-magnesium elements disclosed in Claim 1 of Xiao is 15 to 40%, leaving 60-85 wt% for magnesium. Medlin Decl. Exhibit 2 at ¶ 149. Dr. Medlin also notes that the “Examples of Xiao disclose a range of magnesium from 54.5 wt% - 79.2 wt%.” Id. Thus, Xiao discloses a range of magnesium 54.5-85 wt%, which overlaps and covers much of the claimed range.
<b>Dependent Claim 53</b>	<p><b>Disclosure of Claim Element in Xiao</b></p> <p>53. The magnesium composite as defined in claim 49, wherein said magnesium alloy includes over 50 wt % magnesium, and one or more metals selected from the group consisting of aluminum in an amount of about 0.5-10 wt %, zinc in an amount of about 0.1-6 wt %, zirconium in an amount of about 0.01-3 wt %, manganese in an amount of about 0.15-2 wt %, boron in an amount of about 0.0002-0.04 wt %, and bismuth in amount of about 0.4-0.7 wt %.</p> <p>Claim 1 of Xiao discloses a “magnesium alloy, comprising the components at the weight percentages as follows: Al: 13 to 25%, Zn: 2 to 15%; the remainder is Mg, and a sum of the weight percentages of the components is 100%.” Xiao at Claim 1 (emphasis added). Dr. Medlin opines that a POSITA would have understood that the total range of the non-magnesium elements disclosed in Claim 1 of Xiao is 15 to 40%, leaving 60-85 wt% for magnesium. Medlin Decl. Exhibit 2 at ¶ 149. Dr. Medlin also notes that the “Examples of Xiao disclose a range of magnesium from 54.5 wt% - 79.2 wt%.” Id. Thus, Xiao discloses a range of magnesium 54.5-85 wt%, which overlaps and covers much of the claimed ranges set forth in these claim elements.</p> <p>This claim element would be anticipated solely by the zirconium concentrations falling within the 0.01-6 wt% range. Claim 3 also requires, in addition to a similar zirconium concentration to the Markush claims, a zinc concentration of 0.1-3 wt%.</p>

<b>Dependent Claim 54</b>	<b>Disclosure of Claim Element in Xiao</b>
<p>54. The magnesium composite as defined in claim 49, wherein said magnesium alloy includes over 50 wt % magnesium, and one or more metals selected from the group consisting of aluminum in an amount of about 0.5-10 wt %, zinc in an amount of about 0.1-3 wt %, zirconium in an amount of about 0.01-1 wt %, manganese in an amount of about 0.15-2 wt %, boron in an amount of about 0.0002-0.04 wt %, and bismuth in an amount of about 0.4-0.7 wt %.</p>	<p>Claim 1 of Xiao discloses a “magnesium alloy, comprising the components at the weight percentages as follows: Al: 13 to 25%, Zn: 2 to 15%; the remainder is Mg, and a sum of the weight percentages of the components is 100%.” Xiao at Claim 1 (emphasis added). Dr. Medlin opines that a POSITA would have understood that the total range of the non-magnesium elements disclosed in Claim 1 of Xiao is 15 to 40%, leaving 60-85 wt% for magnesium. Medlin Decl. Exhibit 2 at ¶ 149. Dr. Medlin also notes that the “Examples of Xiao disclose a range of magnesium from 54.5 wt% - 79.2 wt%.” Id. Thus, Xiao discloses a range of magnesium 54.5-85 wt%, which overlaps and covers much of the claimed ranges set forth in these claim elements.</p> <p>This claim element would be anticipated solely by the zirconium concentrations falling within the 0.01-6 wt% range. Claim 3 also requires, in addition to a similar zirconium concentration to the Markush claims, a zinc concentration of 0.1-3 wt%.</p>
<b>Dependent Claim 56</b>	<b>Disclosure of Claim Element in Xiao</b>
<p>56. The magnesium composite as defined in claim 49, wherein said magnesium alloy includes 60-95 wt % magnesium and 0.01-1 wt % zirconium.</p>	<p>Claim 1 of Xiao discloses a “magnesium alloy, comprising the components at the weight percentages as follows: Al: 13 to 25%, Zn: 2 to 15%; the remainder is Mg, and a sum of the weight percentages of the components is 100%.” Xiao at Claim 1 (emphasis added). Dr. Medlin opines that a POSITA would have understood that the total range of the non-magnesium elements disclosed in Claim 1 of Xiao is 15 to 40%, leaving 60-85 wt% for magnesium. Medlin Decl. Exhibit 2 at ¶ 149. Dr. Medlin also notes that the “Examples of Xiao disclose a range of magnesium from 54.5 wt% - 79.2 wt%.” Id. Thus, Xiao discloses a range of magnesium 54.5-85 wt%, which overlaps</p>

	<p>and covers much of the claimed ranges set forth in these claim elements.</p> <p>Claim 2 discloses: a “magnesium alloy, according to Claim 1, further comprising the trace elements at the weight percentages as follows: Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%; and a sum of the weight percentages of the components is 100%.” Id. at Claim 2.</p> <p>These metallic elements are found in the additive materials generally disclosed by Xiao (i.e., intermediate alloys: Al-Fe, Al-Ni, Al-Cu, Al-Ti) (Xiao at ¶ 0023) and particularly described in association with Example 7 of Xiao (Id. at ¶¶ 0057–0060) for which Dr. Medlin calculated for Example 7 of Xiao (Medlin Decl. Exhibit 2 at ¶¶ 113–122) the specific weight percentage (ranges) of each additive material or secondary metal. In particular, Dr. Medlin determined that the additive materials aluminum-iron with 30 wt% iron, aluminum-nickel with 45 wt% nickel, and aluminum-copper with 40 wt% copper also fall well within the range of about 0.05 wt% to 45 wt% of the mixture. Id. ¶ 122. For example, the aluminum-iron additive constitutes 3.3 wt% of the mixture; the aluminum-nickel additive constitutes 1.1 wt% of the mixture; and the aluminum-copper additive constitutes 0.25 wt% of the mixture. Dr. Medlin expressed his opinion that a POSITA in April 2014 would have also recognized that other intermediate alloy compositions would have fallen within the teachings of Example 7 and other disclosures of Xiao resulting in Al-Fe, Al-Ni, and Al-Cu intermediate alloys constituting about 0.05 wt% to 45 wt% of the mixture. Id. at ¶ 125. Accordingly, these claim elements are disclosed by Xiao.</p>
<b>Dependent Claim 57</b>	<b>Disclosure of Claim Element in Xiao</b>

<p>57. The magnesium composite as defined in claim 56, wherein said magnesium alloy further includes 0.05-6 wt % zinc.</p>	<p>Claim 1 of Xiao discloses a “magnesium alloy, comprising the components at the weight percentages as follows: Al: 13 to 25%, Zn: 2 to 15%; the remainder is Mg, and a sum of the weight percentages of the components is 100%.” Xiao at Claim 1 (emphasis added). Dr. Medlin opines that a POSITA would have understood that the total range of the non-magnesium elements disclosed in Claim 1 of Xiao is 15 to 40%, leaving 60-85 wt% for magnesium. Medlin Decl. Exhibit 2 at ¶ 149. Dr. Medlin also notes that the “Examples of Xiao disclose a range of magnesium from 54.5 wt% - 79.2 wt%.” Id. Thus, Xiao discloses a range of magnesium 54.5-85 wt%, which overlaps and covers much of the claimed ranges set forth in these claim elements.</p> <p>Claim 2 discloses: a “magnesium alloy, according to Claim 1, further comprising the trace elements at the weight percentages as follows: Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%; and a sum of the weight percentages of the components is 100%.” Id. at Claim 2.</p> <p>These metallic elements are found in the additive materials generally disclosed by Xiao (i.e., intermediate alloys: Al-Fe, Al-Ni, Al-Cu, Al-Ti) (Xiao at ¶ 0023) and particularly described in association with Example 7 of Xiao (Id. at ¶¶ 0057-0060) for which Dr. Medlin calculated for Example 7 of Xiao (Medlin Decl. Exhibit 2 at ¶¶ 113-122) the specific weight percentage (ranges) of each additive material or secondary metal. In particular, Dr. Medlin determined that the additive materials aluminum-iron with 30 wt% iron, aluminum-nickel with 45 wt% nickel, and aluminum-copper with 40 wt% copper also fall well within the range of about 0.05 wt% to 45 wt% of the mixture. Id. ¶ 122. For example, the aluminum-iron additive constitutes 3.3 wt% of the mixture; the aluminum-nickel</p>
--	---

	<p>additive constitutes 1.1 wt% of the mixture; and the aluminum-copper additive constitutes 0.25 wt% of the mixture. Dr. Medlin expressed his opinion that a POSITA in April 2014 would have also recognized that other intermediate alloy compositions would have fallen within the teachings of Example 7 and other disclosures of Xiao resulting in Al-Fe, Al-Ni, and Al-Cu intermediate alloys constituting about 0.05 wt% to 45 wt% of the mixture. Id. at ¶ 125. Accordingly, these claim elements are disclosed by Xiao.</p>
<b>Dependent Claim 58</b>	<b>Disclosure of Claim Element in Xiao</b>
58. The magnesium composite as defined in claim 49, wherein said magnesium alloy includes 60-95 wt % magnesium, 0.5-10 wt % aluminum, 0.05-6 wt % zinc, and 0.15-2 wt % manganese.	<p>Claim 1 of Xiao discloses a “magnesium alloy, comprising the components at the weight percentages as follows: Al: 13 to 25%, Zn: 2 to 15%; the remainder is Mg, and a sum of the weight percentages of the components is 100%.” Xiao at Claim 1 (emphasis added). Dr. Medlin opines that a POSITA would have understood that the total range of the non-magnesium elements disclosed in Claim 1 of Xiao is 15 to 40%, leaving 60-85 wt% for magnesium. Medlin Decl. Exhibit 2 at ¶ 149. Dr. Medlin also notes that the “Examples of Xiao disclose a range of magnesium from 54.5 wt% - 79.2 wt%.” Id. Thus, Xiao discloses a range of magnesium 54.5-85 wt%, which overlaps and covers much of the claimed ranges set forth in these claim elements.</p> <p>Claim 2 discloses: a “magnesium alloy, according to Claim 1, further comprising the trace elements at the weight percentages as follows: Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%; and a sum of the weight percentages of the components is 100%.” Id. at Claim 2.</p> <p>These metallic elements are found in the additive materials generally disclosed by Xiao (i.e., intermediate alloys: Al-Fe, Al-Ni, Al-Cu, Al-Ti) (Xiao at ¶ 0023) and particularly described in</p>

	association with Example 7 of Xiao (Id. at ¶¶ 0057–0060) for which Dr. Medlin calculated for Example 7 of Xiao (Medlin Decl. Exhibit 2 at ¶¶ 113–122) the specific weight percentage (ranges) of each additive material or secondary metal. In particular, Dr. Medlin determined that the additive materials aluminum-iron with 30 wt% iron, aluminum-nickel with 45 wt% nickel, and aluminum-copper with 40 wt% copper also fall well within the range of about 0.05 wt% to 45 wt% of the mixture. Id. ¶ 122. For example, the aluminum-iron additive constitutes 3.3 wt% of the mixture; the aluminum-nickel additive constitutes 1.1 wt% of the mixture; and the aluminum-copper additive constitutes 0.25 wt% of the mixture. Dr. Medlin expressed his opinion that a POSITA in April 2014 would have also recognized that other intermediate alloy compositions would have fallen within the teachings of Example 7 and other disclosures of Xiao resulting in Al-Fe, Al-Ni, and Al-Cu intermediate alloys constituting about 0.05 wt% to 45 wt% of the mixture. Id. at ¶ 125. Accordingly, these claim elements are disclosed by Xiao.
Dependent Claim 59	Disclosure of Claim Element in Xiao
59. The magnesium composite as defined in claim 49, wherein said magnesium alloy includes over 50 wt % magnesium and one or more metals selected from the group consisting of 0.5-10 wt % aluminum, 0.1-2 wt % zinc, 0.01-1 wt % zirconium, and 0.15-2 wt % manganese.	Claim 1 of Xiao discloses a “magnesium alloy, comprising the components at the weight percentages as follows: Al: 13 to 25%, Zn: 2 to 15%; the remainder is Mg, and a sum of the weight percentages of the components is 100%.” Xiao at Claim 1 (emphasis added). Dr. Medlin opines that a POSITA would have understood that the total range of the non-magnesium elements disclosed in Claim 1 of Xiao is 15 to 40%, leaving 60-85 wt% for magnesium. Medlin Decl. Exhibit 2 at ¶ 149. Dr. Medlin also notes that the “Examples of Xiao disclose a range of magnesium from 54.5 wt% - 79.2 wt%.” Id. Thus, Xiao discloses a range of magnesium 54.5-85 wt%, which overlaps

	<p>and covers much of the claimed ranges set forth in these claim elements.</p> <p>Claim 2 discloses: a “magnesium alloy, according to Claim 1, further comprising the trace elements at the weight percentages as follows: Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%; and a sum of the weight percentages of the components is 100%.” Id. at Claim 2.</p> <p>These metallic elements are found in the additive materials generally disclosed by Xiao (i.e., intermediate alloys: Al-Fe, Al-Ni, Al-Cu, Al-Ti) (Xiao at ¶ 0023) and particularly described in association with Example 7 of Xiao (Id. at ¶¶ 0057–0060) for which Dr. Medlin calculated for Example 7 of Xiao (Medlin Decl. Exhibit 2 at ¶¶ 113–122) the specific weight percentage (ranges) of each additive material or secondary metal. In particular, Dr. Medlin determined that the additive materials aluminum-iron with 30 wt% iron, aluminum-nickel with 45 wt% nickel, and aluminum-copper with 40 wt% copper also fall well within the range of about 0.05 wt% to 45 wt% of the mixture. Id. ¶ 122. For example, the aluminum-iron additive constitutes 3.3 wt% of the mixture; the aluminum-nickel additive constitutes 1.1 wt% of the mixture; and the aluminum-copper additive constitutes 0.25 wt% of the mixture. Dr. Medlin expressed his opinion that a POSITA in April 2014 would have also recognized that other intermediate alloy compositions would have fallen within the teachings of Example 7 and other disclosures of Xiao resulting in Al-Fe, Al-Ni, and Al-Cu intermediate alloys constituting about 0.05 wt% to 45 wt% of the mixture. Id. at ¶ 125. Accordingly, these claim elements are disclosed by Xiao.</p>
<b>Dependent Claim 60</b>	<b>Disclosure of Claim Element in Xiao</b>

<p>60. The magnesium composite as defined in claim 49, wherein said magnesium alloy includes over 50 wt % magnesium, and one or more metals selected from the group consisting of 0.1-3 wt % zinc, 0.01-1 wt % zirconium, 0.05-1 wt % manganese, 0.0002-0.04 wt % boron, and 0.4-0.7 wt % bismuth.</p>	<p>Claim 1 of Xiao discloses a “magnesium alloy, comprising the components at the weight percentages as follows: Al: 13 to 25%, Zn: 2 to 15%; the remainder is Mg, and a sum of the weight percentages of the components is 100%.” Xiao at Claim 1 (emphasis added). Dr. Medlin opines that a POSITA would have understood that the total range of the non-magnesium elements disclosed in Claim 1 of Xiao is 15 to 40%, leaving 60-85 wt% for magnesium. Medlin Decl. Exhibit 2 at ¶ 149. Dr. Medlin also notes that the “Examples of Xiao disclose a range of magnesium from 54.5 wt% - 79.2 wt%.” Id. Thus, Xiao discloses a range of magnesium 54.5-85 wt%, which overlaps and covers much of the claimed ranges set forth in these claim elements.</p> <p>Claim 2 discloses: a “magnesium alloy, according to Claim 1, further comprising the trace elements at the weight percentages as follows: Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%; and a sum of the weight percentages of the components is 100%.” Id. at Claim 2. These metallic elements are found in the additive materials generally disclosed by Xiao (i.e., intermediate alloys: Al-Fe, Al-Ni, Al-Cu, Al-Ti) (Xiao at ¶ 0023) and particularly described in association with Example 7 of Xiao (Id. at ¶¶ 0057-0060) for which Dr. Medlin calculated for Example 7 of Xiao (Medlin Decl. Exhibit 2 at ¶¶ 113-122) the specific weight percentage (ranges) of each additive material or secondary metal. In particular, Dr. Medlin determined that the additive materials aluminum-iron with 30 wt% iron, aluminum-nickel with 45 wt% nickel, and aluminum-copper with 40 wt% copper also fall well within the range of about 0.05 wt% to 45 wt% of the mixture. Id. ¶ 122. For example, the aluminum-iron additive constitutes 3.3 wt% of the mixture; the aluminum-nickel</p>
--	--

	<p>additive constitutes 1.1 wt% of the mixture; and the aluminum-copper additive constitutes 0.25 wt% of the mixture. Dr. Medlin expressed his opinion that a POSITA in April 2014 would have also recognized that other intermediate alloy compositions would have fallen within the teachings of Example 7 and other disclosures of Xiao resulting in Al-Fe, Al-Ni, and Al-Cu intermediate alloys constituting about 0.05 wt% to 45 wt% of the mixture. Id. at ¶ 125. Accordingly, these claim elements are disclosed by Xiao.</p>
<b>Dependent Claim 61</b>	<b>Disclosure of Claim Element in Xiao</b>
61. The magnesium composite as defined in claim 49, wherein said additive material includes nickel,	<p>Xiao discloses a “magnesium alloy...and further adds elements of Fe, Cu, Ni, and Ag which can enhance the corrosion performance of the magnesium alloy...” Xiao at ¶ 0026, ll. 1–9. See Declaration of Dr. Dana Medlin (Medlin Decl. Exhibit 2, e.g., at ¶ 104-105 (Claim 1)).</p> <p>Xiao teaches the use of a variety of metals as additive materials. See, e.g., Xiao at ¶ 0026. For instance, Xiao claims that the novel magnesium alloy may “further compris[e] trace elements at the weight percentages as follows: Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%...”Id. at Claim 2. In addition, Example 7 of Xiao particularly discloses intermediate alloys Al-Fe, Al-Ni, Al-Ag, Al-Cu, Al-Zr, Al-Ti and Zinc as additive materials. Id. at ¶ 0060. A POSITA in April 2014 would have understood that all of these intermediate alloys contain metal, including nickel, among other metals. Medlin Decl. Exhibit 2 at ¶ 145. Thus, Xiao discloses a number of metal additives, including nickel, and hence fully discloses all of the limitations of these claim elements.</p>
said nickel constitutes about 0.05-35 wt % of said magnesium composite,	Claim 1 of Xiao discloses: a “magnesium alloy, comprising the components at the weight percentages as follows: Al: 13 to 25%, Zn: 2 to 15%, the remainder is Mg, and a sum of the

	<p>weight percentages of the components is 100%.” Xiao at Claim 1. And Claim 2 discloses: a “magnesium alloy, according to Claim 1, further comprising the trace elements at the weight percentages as follows: Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%; and a sum of the weight percentages of the components is 100%.” Id. at Claim 2.</p> <p>These metallic elements are found in the additive materials generally disclosed by Xiao (i.e., intermediate alloys: Al-Fe, Al-Ni, Al-Cu, Al-Ti) (Xiao at ¶ 0023) and particularly described in association with Example 7 of Xiao (Id. at ¶¶ 0057–0060) for which Dr. Medlin calculated for Example 7 of Xiao (Medlin Decl. Exhibit 2 at ¶¶ 113–122) the specific weight percentage (ranges) of each additive material or secondary. In particular, Dr. Medlin determined that the additive materials aluminum-iron with 30 wt% iron, aluminum-nickel with 45 wt% nickel, and aluminum-copper with 40 wt% copper also fall well within the range of about 0.05 wt% to 45 wt% of the mixture. Id. ¶ 122. For example, the aluminum-iron additive constitutes 3.3 wt% of the mixture; the aluminum-nickel additive constitutes 1.1 wt% of the mixture; and the aluminum-copper additive constitutes 0.25 wt% of the mixture. Dr. Medlin expressed his opinion that a POSITA in April 2014 would have also recognized that other intermediate alloy compositions would have fallen within the teachings of Example 7 and other disclosures of Xiao resulting in Al-Fe, Al-Ni, and Al-Cu intermediate alloys constituting about 0.05 wt% to 45 wt% of the mixture. Id. at ¶ 125. Accordingly, this claim element was disclosed by Xiao.</p>
said nickel forms galvanically-active in situ	Xiao discloses a novel magnesium alloy with high aluminum and zinc content further adding elements Fe, Cu, Ni and Ag which can form intermetallics and enhance the corrosion

<p>precipitate in said magnesium composite.</p>	<p>performance of the magnesium alloy. Xiao at ¶ 0026. Xiao explains the corrosion mechanism within a magnesium alloy in terms of a cathode phase, which Xiao explains includes “the <math>\beta</math> (<math>Mg_{17}Al_{12}</math>) phase and eutectic phase on the grain boundary of the magnesium alloy,” and “the magnesium matrix <math>\alpha</math> phase which functions as an anode phase, so that the matrix and the grain boundary of the magnesium alloy form a large amount of micro-batteries, which [micro-batteries] greatly accelerate the corrosion decomposition of magnesium alloy.” Id.</p> <p>A POSITA in August 2014 would have understood this disclosure of “intermetallic composite micro-particles” to be a reference to intermetallic precipitate. Medlin Decl. Exhibit 2 at ¶ 107.</p> <p>A POSITA reading the Xiao reference in April 2014 would have recognized that the precipitation of galvanically-active intermetallic phases (“micro-batteries”) are naturally and necessarily generated by the in situ reaction between magnesium and any and all of the disclosed metal additives forming in situ precipitate. Medlin Decl. Exhibit 2, at ¶ 108. Thus, these claim elements were disclosed by Xiao.</p> <p>These elements are among the claimed “secondary metals” disclosed by Xiao. Thus, all of the limitations of this claim element are disclosed by Xiao.</p>
<p><b>Dependent Claim 62</b></p> <p>62. The magnesium composite as defined in claim 61, wherein said nickel</p>	<p><b>Disclosure of Claim Element in Xiao</b></p> <p>Claim 1 of Xiao discloses: a “magnesium alloy, comprising the components at the weight percentages as follows: Al: 13 to 25%, Zn: 2 to 15%, the remainder is Mg, and a sum of the weight percentages of the components is 100%.” Xiao at Claim</p>

constitutes about 3-7 wt % of said magnesium composite.	<p>1. And Claim 2 discloses: a “magnesium alloy, according to Claim 1, further comprising the trace elements at the weight percentages as follows: Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%; and a sum of the weight percentages of the components is 100%.” Id. at Claim 2.</p> <p>These metallic elements are found in the additive materials generally disclosed by Xiao (i.e., intermediate alloys: Al-Fe, Al-Ni, Al-Cu, Al-Ti) (Xiao at ¶ 0023) and particularly described in association with Example 7 of Xiao (Id. at ¶¶ 0057–0060) for which Dr. Medlin calculated for Example 7 of Xiao (Medlin Decl. Exhibit 2 at ¶¶ 113–122) the specific weight percentage (ranges) of each additive material or secondary metal. In particular, Dr. Medlin determined that the additive materials aluminum-iron with 30 wt% iron, aluminum-nickel with 45 wt% nickel, and aluminum-copper with 40 wt% copper also fall well within the range of about 0.05 wt% to 45 wt% of the mixture. Id. ¶ 122. For example, the aluminum-iron additive constitutes 3.3 wt% of the mixture; the aluminum-nickel additive constitutes 1.1 wt% of the mixture; and the aluminum-copper additive constitutes 0.25 wt% of the mixture. Dr. Medlin expressed his opinion that a POSITA in April 2014 would have also recognized that other intermediate alloy compositions would have fallen within the teachings of Example 7 and other disclosures of Xiao resulting in Al-Fe, Al-Ni, and Al-Cu intermediate alloys constituting about 0.05 wt% to 45 wt% of the mixture. Id. at ¶ 125. Accordingly, this claim element was disclosed by Xiao.</p>
<b>Dependent Claim 64</b>	<b>Disclosure of Claim Element in Xiao</b>

<p>64. The magnesium composite as defined in claim 49, wherein said additive material includes copper,</p>	<p>Xiao discloses a “magnesium alloy...and further adds elements of Fe, Cu, Ni, and Ag which can enhance the corrosion performance of the magnesium alloy...” Xiao at ¶ 0026, ll. 1–9. See Declaration of Dr. Dana Medlin (Medlin Decl. Exhibit 2, e.g., at ¶ 104-105 (Claim 1)).</p> <p>Xiao teaches the use of a variety of metals as additive materials. See, e.g., Xiao at ¶ 0026. For instance, Xiao claims that the novel magnesium alloy may “further compris[e] trace elements at the weight percentages as follows: Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%...” Id. at Claim 2. In addition, Example 7 of Xiao particularly discloses intermediate alloys Al-Fe, Al-Ni, Al-Ag, Al-Cu, Al-Zr, Al-Ti and Zinc as additive materials. Id. at ¶ 0060. A POSITA in April 2014 would have understood that all of these intermediate alloys contain metal, including, in particular, copper, among other metals. Medlin Decl. Exhibit 2 at ¶ 145. Thus, Xiao discloses a number of metal additives, including copper, and hence fully discloses all of the limitations of this claim element.</p>
<p>said copper constitutes about 0.05-35 wt % of said magnesium composite,</p>	<p>Claim 1 of Xiao discloses: a “magnesium alloy, comprising the components at the weight percentages as follows: Al: 13 to 25%, Zn: 2 to 15%, the remainder is Mg, and a sum of the weight percentages of the components is 100%.” Xiao at Claim 1. And Claim 2 discloses: a “magnesium alloy, according to Claim 1, further comprising the trace elements at the weight percentages as follows: Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%; and a sum of the weight percentages of the components is 100%.” Id. at Claim 2.</p> <p>These metallic elements are found in the additive materials generally disclosed by Xiao (i.e., intermediate alloys: Al-Fe, Al-Ni, Al-Cu, Al-Ti) (Xiao at ¶ 0023) and particularly described in</p>

	<p>association with Example 7 of Xiao (Id. at ¶¶ 0057–0060) for which Dr. Medlin calculated for Example 7 of Xiao (Medlin Decl. Exhibit 2 at ¶¶ 113–122) the specific weight percentage (ranges) of each additive material or secondary metal. In particular, Dr. Medlin determined that the additive materials aluminum-iron with 30 wt% iron, aluminum- nickel with 45 wt% nickel, and aluminum-copper with 40 wt% copper also fall well within the range of about 0.05 wt% to 45 wt% of the mixture. Id. ¶ 122. For example, the aluminum-iron additive constitutes 3.3 wt% of the mixture; the aluminum-nickel additive constitutes 1.1 wt% of the mixture; and the aluminum-copper additive constitutes 0.25 wt% of the mixture. Dr. Medlin expressed his opinion that a POSITA in April 2014 would have also recognized that other intermediate alloy compositions would have fallen within the teachings of Example 7 and other disclosures of Xiao resulting in Al-Fe, Al-Ni, and Al-Cu intermediate alloys constituting about 0.05 wt% to 45 wt% of the mixture. Id. at ¶ 125. Accordingly, this claim element is disclosed by Xiao.</p>
said copper forms the galvanically-active in situ precipitate in said magnesium composite.	Xiao discloses a novel magnesium alloy with high aluminum and zinc content further adding elements Fe, Cu, Ni and Ag which can form intermetallics and enhance the corrosion performance of the magnesium alloy. Xiao at ¶ 0026. Xiao explains the corrosion mechanism within a magnesium alloy in terms of a cathode phase, which Xiao explains includes “the $\beta$ ( $Mg_{17}Al_{12}$ ) phase and eutectic phase on the grain boundary of the magnesium alloy,” and “the magnesium matrix $\alpha$ phase which functions as an anode phase, so that the matrix and the grain boundary of the magnesium alloy form a large amount of micro-batteries, which [micro-batteries] greatly accelerate the corrosion decomposition of magnesium alloy.” Id.

	These elements are among the claimed “secondary metals” disclosed by Xiao. Thus, all of the limitations of this claim element are disclosed by Xiao.
<b>Dependent Claim 66</b>	<b>Disclosure of Claim Element in Xiao</b>
66. The magnesium composite as defined in claim 49, wherein a dissolution rate of said magnesium composite is about 5-325 mg/cm <sup>2</sup> /hr. in 3 wt % KCl water mixture at 90°C.	<p>A POSITA in April 2014 would have recognized that Xiao disclosed a table of dissolution rates (“decomposition rates”) because the units of measurement are the units used by POSITAs to measure dissolution rates. Medlin Decl. Exhibit 2 at ¶ 129. The disclosed table provides seven examples (excluding the comparative [prior art] example). Xiao at ¶ 0064. Xiao provides dissolution rates in g/cm<sup>2</sup>/hr, while the claims of the ’653 Patent express dissolution rates in mg/cm<sup>2</sup>/hr. A POSITA would have known converting between g/cm<sup>2</sup>/hr to mg/cm<sup>2</sup>/hr, merely requires multiplication by 1,000. Medlin Decl. Exhibit 2 at ¶ 130.</p> <p>Thus, each of Xiao’s seven examples (excluding the comparative [prior art] example) has a dissolution rate within the claimed range of “about 5-325 mg/cm<sup>2</sup>/hr in 3 wt.% KCl water” at both 70°C or 93°C (with Example 7 being more than seven times that floor), Dr. Medlin opines that a POSITA in April 2014 would have recognized that the dissolution rate of the examples in Xiao would necessarily have been well above the claimed floor of “about 5” mg/cm<sup>2</sup>/hr at the claimed 90°C. Id. at ¶ 132.</p>
<b>Dependent Claim 67</b>	<b>Disclosure of Claim Element in Xiao</b>
67. The magnesium composite as defined in claim 49, wherein a dissolution rate of said magnesium composite is at least 45 mg/cm <sup>2</sup> /hr. in 3 wt % KCl water mixture at	A POSITA in April 2014 would have recognized that Xiao disclosed a table of dissolution rates (“decomposition rates”) because the units of measurement are the units used by POSITAs to measure dissolution rates. Medlin Decl. Exhibit 2 at ¶ 129. The disclosed table provides seven examples (excluding the comparative [prior art] example). Xiao at ¶ 0064.

<p>90°C. and up to 325 mg/cm<sup>2</sup>/hr. in 3 wt % KCl water mixture at 90°C.</p>	<p>Xiao provides dissolution rates in g/cm<sup>2</sup>/hr, while the claims of the '653 Patent express dissolution rates in mg/cm<sup>2</sup>/hr. A POSITA would have known converting between g/cm<sup>2</sup>/hr to mg/cm<sup>2</sup>/hr, merely requires multiplication by 1,000. Medlin Decl. Exhibit 2 at ¶ 130.</p> <p>Six of the seven examples (excluding the comparative [prior art] example) provided in Xiao therefore discloses a dissolution rate of “at least 45 mg/cm<sup>2</sup>/hr in 3 wt % KCl water” and “up to 325 mg/cm<sup>2</sup>/hr in 3 wt % KCl water” at 93°C. Given that the dissolution rates of Xiao slightly above (i.e., 3°C above) 90°C are almost all above the claimed floor of “at least 45 mg/cm<sup>2</sup>/hr”. As such and per Dr. Medlin’s opinion, a POSITA in April 2014 would have expected the dissolution rate of the examples in Xiao at 90°C to also be necessarily above the claimed floor of “at least 45 mg/cm<sup>2</sup>/hr.” Medlin Decl. Exhibit 2 at ¶ 205. Thus, this element is disclosed by Xiao.</p>
---	---

Dependent Claim 69	Disclosure of Claim Element in Xiao
<p>69. The magnesium composite as defined in claim 49, wherein said additive material has a melting point temperature that is 100°C. greater than a melting temperature of said magnesium or magnesium alloy.</p>	<p>Xiao discloses a “magnesium alloy...and further adds elements of Fe, Cu, Ni, and Ag which can enhance the corrosion performance of the magnesium alloy...” Xiao at ¶ 0026, ll. 1–9. See Declaration of Dr. Dana Medlin (Medlin Decl. Exhibit 2, e.g., at ¶ 104-105 (Claim 1)).</p> <p>As Dr. Medlin opines, a POSITA in April 2014 would have understood “melting temperature” in this claim element to be a reference to the smelting temperature or temperature of the melt when the additive material is being added to the magnesium or magnesium alloy, not the “melting point temperature” as that measurement is used for the additive material. Medlin Decl. Exhibit 2 at ¶ 235.</p> <p>Xiao generally discloses “loading pure magnesium and pure aluminum into a smelting furnace and increasing the</p>

temperature to 700 to 730 °C, next loading pure zinc and an [sic] intermediate alloys . . . into a resulting magnesium-aluminum alloy melt after melting and increasing the temperature to 740 to 780°C . . . .” Xiao at ¶¶ 0021–0022; see also Id. at Claim 5 (“the melting temperature of the pure magnesium and pure aluminum is from 700 to 730 °C.”)

Examples 1–7 of Xiao teach a range of smelting temperatures for the magnesium-aluminum alloy from 700°C (Example 1) to 730°C (Examples 2, 5, and 7). Xiao ¶¶ 0034–0060.

The additive materials noted above are one or more of the intermediate alloys: Al-Fe, Al-Ni, Al-Cu, Al-Ag, Al-Zr, Al-Ti. Id. at ¶ 0023. A POSITA using typical phase diagrams for each of these additive materials would be able to readily determine the melting point temperature. Medlin Decl. Exhibit 2, e.g., at ¶¶ 50- 52.

Dr. Medlin reviewed phase diagrams and opined that a POSITA in April 2014 would have recognized that Al-Fe had a variable melting point temperature ranging from 1538°C (pure iron) down to 652°C for a eutectic point near 99 wt% aluminum; Al-Ni had a variable melting point temperature that ranges from 1640°C down to 640°C (depending upon the alloy mixture); and Al-Cu had a variable melting point temperature ranging from 548°C up to 1084°C (depending upon the alloy mixture). Medlin Decl. Exhibit 2 at ¶ 239.

Example 7 of Xiao specifically discloses a melting or smelting temperature of 730°C. Xiao at ¶ 0060. Dr. Medlin specifically analyzed Example 7 of Xiao to determine (using particular intermediate alloy compositions) the melting point temperatures of the intermediate alloys added. Medlin Decl. Exhibit 2 at ¶ 240. It is thus Dr. Medline’s opinion that a POSITA in April 2014 would have recognized from Example 7

	of Xiao that at least an aluminum-nickel additive having 45 wt% nickel has a melting point temperature of 854°C which is 100°C greater than the melting or smelting temperatures of the magnesium aluminum alloy used in Example 7 (i.e., 730°C) and otherwise taught generally by Xiao (i.e., 700-730°C). Id. at ¶ 381. A POSITA in April 2014 would have also recognized that other intermediate alloy compositions could have fallen within the teachings of Example 7 and other disclosures of Xiao and resulted in Al-Fe, Al-Ni, and Al-Cu intermediate alloys that would have had melting point temperatures that were 100°C. greater than the smelting temperatures of the magnesium alloy generally taught by Xiao (i.e., 700- 730°C). Id. Thus, this claim element was disclosed by Xiao.
Dependent Claim 70	Disclosure of Claim Element in Xiao
70. The magnesium composite as defined in claim 49, wherein said magnesium composite is at least partially included in a down hole well component,	Xiao discloses a “magnesium alloy...and further adds elements of Fe, Cu, Ni, and Ag which can enhance the corrosion performance of the magnesium alloy...” Xiao at ¶ 0026, ll. 1–9. See Declaration of Dr. Dana Medlin (Medlin Decl. Exhibit 2, e.g., at ¶ 104-105 (Claim 1)).  Xiao discloses that the cast magnesium alloy “can be used as a tripping ball material for a multi-stage sliding sleeve staged-fracturing technique.” ¶ 0001. A POSITA would have understood that a tripping ball was a type of tool component in an oil well drilling operation. Medlin Decl. Exhibit 2 at ¶ 148. Thus, these claim elements were disclosed by Xiao.
said down hole well component including one or more components selected from the group consisting of a sleeve, frac ball, hydraulic	Xiao discloses that the cast magnesium alloy “can be used as a tripping ball material for a multi-stage sliding sleeve staged-fracturing technique.” Xiao at ¶ 0001. A POSITA would have understood that a tripping ball was a type of tool component in an oil well drilling operation. Medlin Decl. Exhibit 2 at ¶ 148.

actuating tooling, tube, valve, valve component, or plug.	
<b>Dependent Claim 72</b>	<b>Disclosure of Claim Element in Xiao</b>
72. The magnesium composite as defined in claim 49, wherein said additive material is a metal or metal alloy.	Xiao discloses a “magnesium alloy...and further adds elements of Fe, Cu, Ni, and Ag which can enhance the corrosion performance of the magnesium alloy...” Xiao at ¶ 0026, ll. 1–9. See Declaration of Dr. Dana Medlin (Medlin Decl. Exhibit 2, e.g., at ¶ 104-105 (Claim 1)).  Xiao teaches the use of a variety of metals as additive materials. See, e.g., Xiao at ¶ 0026. For instance, Xiao claims that the magnesium alloy may “further compris[e] trace elements at the weight percentages as follows: Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%...” Id. at Claim 2. In addition, Example 7 of Xiao particularly discloses intermediate alloys Al-Fe, Al-Ni, Al-Ag, Al-Cu, Al-Zr, Al-Ti and Zinc as additive materials. Id. at ¶ 0060. A POSITA in April 2014 would have understood that all of these additive materials contain metal(s). Medlin Decl. Exhibit 2 at ¶ 145. Accordingly, Example 7 of Xiao discloses this claim element in accordance with Patent Owner’s definition of “additive material.”
<b>Independent Claim 73</b>	<b>Disclosure of Claim Element in Xiao</b>
73. A dissolvable magnesium composite for use in a ball or other tool component in a well drilling or completion operation,	Xiao discloses a “magnesium alloy...and further adds elements of Fe, Cu, Ni, and Ag which can enhance the corrosion performance of the magnesium alloy...” Xiao at ¶ 0026, ll. 1–9. See Declaration of Dr. Dana Medlin (Medlin Decl. Exhibit 2, e.g., at ¶ 104-105 (Claim 1)).  Xiao discloses that the cast magnesium alloy “can be used as a tripping ball material for a multi-stage sliding sleeve staged-fracturing technique.” ¶ 0001. A POSITA would have understood that a tripping ball was a type of tool component in

	an oil well drilling operation. Medlin Decl. Exhibit 2 at ¶ 148. Thus, these claim elements were disclosed by Xiao.
said dissolvable magnesium composite includes in situ precipitation of galvanically-active intermetallic phases to enable controlled dissolution of said magnesium composite,	Xiao discloses a novel magnesium alloy with high aluminum and zinc content further adding elements Fe, Cu, Ni and Ag which can form intermetallics and enhance the corrosion performance of the magnesium alloy. Xiao explains the corrosion mechanism within a magnesium alloy in terms of a cathode phase, which Xiao explains includes “the $\beta$ ( $Mg_{17}Al_{12}$ ) phase and eutectic phase on the grain boundary of the magnesium alloy,” and “the magnesium matrix $\alpha$ phase which functions as an anode phase, so that the matrix and the grain boundary of the magnesium alloy form a large amount of micro-batteries, which [micro-batteries] greatly accelerate the corrosion decomposition of magnesium alloy.” Xiao at ¶ 0026. A POSITA in August 2014 would have understood this disclosure in Xiao (¶ 0026) of “intermetallic composite micro-particles” to be a reference to intermetallic precipitate. Medlin Decl. Exhibit 2 at ¶ 107.
	A POSITA reading the Xiao reference in April 2014 would have recognized that the precipitation of galvanically-active intermetallic phases (“micro-batteries”) are naturally and necessarily generated by the in situ reaction between magnesium and any and all of the disclosed metal additives forming in situ precipitate. Medlin Decl. Exhibit 2, at ¶ 108.
said magnesium composite comprising a mixture of magnesium or a magnesium alloy and an additive material,	Xiao discloses “first loading pure magnesium and pure aluminum into a smelting furnace . . . next loading pure zinc and an [sic] intermediate alloys of trace element components into a resulting magnesium-aluminum alloy melt after melting.” Xiao, at ¶ 0022. Xiao discloses that “Al-Fe . . . Al-Ni . . . Al-Cu . . . Al-Ag . . . Al-Zr . . . and . . . Al-Ti intermediate alloy[s], are heated to

	<p>dry and then added to the magnesium-aluminum alloy melt.” Id. at ¶ 0023. These intermediate alloys meet the Patent Owner’s definition of “additive material,” i.e., “a material that is added.” Medlin Decl. Exhibit 2, at ¶ 110. The addition of these intermediate alloys to the melted magnesium alloy necessarily results in a mixture of the additive material and magnesium alloy. Id. at ¶ 111.</p>
said additive material constituting about 0.05 wt % of said mixture,	<p>Claim 1 of Xiao discloses: a “magnesium alloy, comprising the components at the weight percentages as follows: Al: 13 to 25%, Zn: 2 to 15%, the remainder is Mg, and a sum of the weight percentages of the components is 100%.” Xiao at Claim 1. And Claim 2 discloses: a “magnesium alloy, according to Claim 1, further comprising the trace elements at the weight percentages as follows: Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%; and a sum of the weight percentages of the components is 100%.” Id. at Claim 2.</p> <p>These metallic elements are found in the additive materials generally disclosed by Xiao (i.e., intermediate alloys: Al-Fe, Al-Ni, Al-Cu, Al-Ti) (Xiao at ¶ 0023) and particularly described in association with Example 7 of Xiao (Id. at ¶¶ 0057–0060) for which Dr. Medlin calculated for Example 7 of Xiao (Medlin Decl. Exhibit 2 at ¶¶ 113–122) the specific weight percentage (ranges) of each additive material or secondary metal. In particular, Dr. Medlin determined that the additive materials aluminum-iron with 30 wt% iron, aluminum-nickel with 45 wt% nickel, and aluminum-copper with 40 wt% copper also fall well within the range of about 0.05 wt% to 45 wt% of the mixture. Id. ¶ 122. For example, the aluminum-iron additive constitutes 3.3 wt% of the mixture; the aluminum-nickel additive constitutes 1.1 wt% of the mixture; and the aluminum-</p>

	copper additive constitutes 0.25 wt% of the mixture. Dr. Medlin expressed his opinion that a POSITA in April 2014 would have also recognized that other intermediate alloy compositions would have fallen within the teachings of Example 7 and other disclosures of Xiao resulting in Al-Fe, Al-Ni, and Al-Cu intermediate alloys constituting about 0.05 wt% to 45 wt% of the mixture. Id. at ¶ 125. Accordingly, these claim elements are disclosed by Xiao.
said additive material is a metal or metal alloy,	Xiao discloses a “magnesium alloy...and further adds elements of Fe, Cu, Ni, and Ag which can enhance the corrosion performance of the magnesium alloy...” Xiao at ¶ 0026, ll. 1–9. See Declaration of Dr. Dana Medlin (Medlin Decl. Exhibit 2, e.g., at ¶ 104-105 (Claim 1)).  Xiao teaches the use of a variety of metals as additive materials. See, e.g., Xiao at ¶ 0026. For instance, Xiao claims that the magnesium alloy may “further compris[e] trace elements at the weight percentages as follows: Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%. . .” Id. at Claim 2. In addition, Example 7 of Xiao particularly discloses intermediate alloys Al-Fe, Al-Ni, Al-Ag, Al-Cu, Al-Zr, Al-Ti and Zinc as additive materials. Id. at ¶ 0060. A POSITA in April 2014 would have understood that all of these additive materials contain metal(s). Medlin Decl. Exhibit 2 at ¶ 145. Accordingly, Example 7 of Xiao discloses this claim element in accordance with Patent Owner’s definition of “additive material.”
said additive material includes one or more metals selected from the group consisting of copper, nickel, titanium, iron, silicon, and cobalt,	Xiao teaches the use of a variety of metals as additive materials. See, e.g., Xiao at ¶ 0026. For instance, Xiao claims that the novel magnesium alloy may “further compris[e] trace elements at the weight percentages as follows: Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%. . .”Id. at Claim 2. In addition, Example 7 of Xiao particularly discloses

	intermediate alloys Al-Fe, Al-Ni, Al-Ag, Al-Cu, Al-Zr, Al-Ti and Zinc as additive materials. Id. at ¶ 0060. A POSITA in April 2014 would have understood that all of these intermediate alloys contain metal, including, in particular, copper, nickel, and iron, among other metals. Medlin Decl. Exhibit 2 at ¶ 145. Thus, Xiao discloses a number of metal additives, including copper, nickel, and iron and, hence fully discloses all of the limitations of these claim elements.
said magnesium composite including in situ precipitation of galvanically-active intermetallic phases that include said additive material,	Xiao discloses a novel magnesium alloy with high aluminum and zinc content further adding elements Fe, Cu, Ni and Ag which can form intermetallics and enhance the corrosion performance of the magnesium alloy. Xiao at ¶ 0026. Xiao explains the corrosion mechanism within a magnesium alloy in terms of a cathode phase, which Xiao explains includes “the $\beta$ ( $Mg_{17}Al_{12}$ ) phase and eutectic phase on the grain boundary of the magnesium alloy,” and “the magnesium matrix $\alpha$ phase which functions as an anode phase, so that the matrix and the grain boundary of the magnesium alloy form a large amount of micro-batteries, which [micro- batteries] greatly accelerate the corrosion decomposition of magnesium alloy.” Id. As Dr. Medlin explains, a POSITA in August 2014 would have understood this disclosure in Xiao of “intermetallic composite micro-particles” (¶ 0026) to be a reference to intermetallic precipitate. Medlin Decl. Exhibit 2 at ¶ 256. As noted above, at least Fe, Cu, and Ni are “additive materials.” Consequently, the in-situ precipitation of galvanically- active intermetallic phases will include one or more of these additive materials. Thus, Xiao discloses all of these claim limitations.
said additive material located in sufficient quantities in said galvanically-active	A POSITA would understand that the galvanically-active intermetallic phases (“composite micro-particles”) have a morphology that enhances the corrosion dissolution rate of the

<p>intermetallic phases so as to obtain a composition and morphology of said galvanically-active intermetallic phases such that a galvanic corrosion rate along said galvanically-active intermetallic phases causes said magnesium composite to have a dissolution rate of at least at least 5 mg/cm<sup>2</sup>/hr. in 3 wt % KCl water mixture at 90°C.</p>	<p>alloy due to a larger number of smaller particles throughout the microstructure. Medlin Decl. Exhibit 2 at ¶ 229. A POSITA would also understand the relationship between “composite micro-particles” and morphology of the galvanically-active intermetallic phases. Id.</p> <p>With respect to the “sufficient quantities” limitation, other claim limitations in Claims 49 and 73 define the weight percentage for the additive material as 0.05-45 wt% and about 0.05 wt%, respectively, which limitations are met by the teaching of Xiao with respect to four metals. In particular, Xiao discloses that “[e]lements such as Fe, Cu, Ni, Ag, etc., in the magnesium alloy can form a large number of intermetallic composite micro-particles, which can improve the corrosion performance of the magnesium alloy, thereby promoting the decomposition of the magnesium alloy.” Id. ¶ 0026. And the magnesium alloy may “further compris[e] trace elements at the weight percentages as follows: Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%;...” Id. at Claim 2. As such, Xiao discloses four metals (i.e., Fe, Cu, Ni, and Ti) included within the list of “additive material” set forth in Claim Element M, supra. The claimed weight % quantities of the “additive material” of Claim Element I, supra, also overlap with the weight % ranges of Xiao’s “trace elements”. As such, Claim Element P of claims 49 and 73-- that the “additive materials” are located in “sufficient quantities”--is fully disclosed by Xiao’s Claim 2 disclosure of “trace elements at the weight percentages as follows: Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%...” Id. As such, Xiao’s four metal “trace elements” are present in “sufficient quantities” to meet Claim Element P of claims 49 and 73.</p>
--	---

	<p>A POSITA in April 2014 would have recognized that Xiao disclosed a table of dissolution rates (“decomposition rates”) because the units of measurement are the units used by POSITAs to measure dissolution rates. Medlin Decl. Exhibit 2 at ¶ 129. The disclosed table provides seven examples (excluding the comparative [prior art] example). Xiao at ¶ 0064. Xiao provides dissolution rates in g/cm<sup>2</sup>/hr, while the claims of the ’653 Patent express dissolution rates in mg/cm<sup>2</sup>/hr. A POSITA would have known converting between g/cm<sup>2</sup>/hr to mg/cm<sup>2</sup>/hr, merely requires multiplication by 1,000. Medlin Decl. Exhibit 2 at ¶ 130.</p> <p>Thus, each of Xiao’s seven examples (excluding the comparative [prior art] example) has a dissolution rate of at least three times the claimed “at least 5 mg/cm<sup>2</sup>/hr in 3 wt.% KCl water” at both 70°C or 93°C (with Example 7 being more than seven times that floor), Dr. Medlin opines that a POSITA in April 2014 would have recognized that the dissolution rate of the examples in Xiao would necessarily have been well above the claimed floor of “at least of 5 mg/cm<sup>2</sup>/hr” at the claimed 90°C. Id. at ¶ 132.</p>
--	--

**A. Ground II: Claims 8, 25–28, 33–36, 55, and 74–76 are anticipated, or at least made obvious, by Xiao**

**i. Anticipation and/or Single-Reference Obviousness**

Claims 8, 25, 33, 55, and 74 each contain at least one limitation that, if not outright disclosed by Xiao, is “almost” disclosed by Xiao, or it is disclosed by combining the prior art disclosed in Xiao with the teachings of Xiao. However, for anticipation a prior art reference must “disclose all elements of the claim within the four corners of the document,” and it must “disclose those elements ‘arranged as in the claim.’” *NetMoneyIN, Inc. v. VeriSign, Inc.*, 545 F.3d 1359,

1369 (Fed. Cir. 2008) (quoting *Connell v. Sears, Roebuck & Co.*, 722 F.2d 1542, 1548 (Fed. Cir. 1983)). Still, a reference can anticipate a claim even if it “does not expressly spell out” all the limitations arranged or combined as in the claim, if a person of skill in the art, reading the reference, would ‘at once envisage’ the claimed arrangement or combination.” *Kennametal, Inc. v. Ingersoll Cutting Tool Co.*, 780 F.3d 1376, 1381 (Fed. Cir. 2015) (quoting *In re Petering*, 301 F.2d 676, 681 (CCPA)). Moreover, a disclosure that anticipates also renders the claim invalid because “anticipation is the epitome of obviousness.” *Connell*, 722 F.2d at 1548 (quoting *In re Fracalossi*, 681 F.2d 792, 794 (CCPA)). Thus, “a prior art disclosure that ‘almost’ meets the standard may render the claim invalid under § 103; it does not ‘anticipate.’” *Id.* With respect to the Challenged Claims of Ground II, if Xiao does not explicitly fully disclose the remaining elements of these claims (as addressed below), it almost does and, thus, renders each of those claims invalid as obvious.

**ii. Independent Claims 25, 33, and 74 and Dependent Claims 8, 26–28, 34–36, 55, and 75–76 are anticipated, or at least made obvious, by Xiao**

<u>'653 Patent</u> <u>Claims/Elements</u>	<u>Anticipated, Or at Least Made Obvious, by Disclosures of</u> <u>CN 103343271 A Xiao et al. (“Xiao”) and Prior Art</u> <u>Magnesium Disclosed in Xiao</u>
<b>Dependent Claim 8</b>	<b>Disclosure of Claim Element in Xiao and/or Prior Art</b> <b>Magnesium Disclosed in Xiao</b>
8. The magnesium composite as defined in claim 1, wherein said magnesium composite is subjected to a surface treatment to improve a surface hardness of said magnesium composite,	As set forth in Ground I, <i>supra</i> , Xiao already discloses each and every element of the magnesium composition of Claim 1. Xiao does not claim, nor disclose in any novel embodiment, subjecting any of its novel magnesium composites to surface treatments (i.e. peening, heat treatment, aluminizing or combinations thereof) nor does it teach that surface treatment

	<p>would achieve any of the benefits set forth in Claim 8 of the ‘653 Patent.</p> <p>However, Xiao discloses that a U.S. Patent Publication teaches a method “for preparing a composite material having a decomposable multilayer coated core-shell structure (U.S. Patent No. US2011/0132143A1, 2011). ¶ 0002. It is explicitly noted in Xiao that this U.S. patent publication discloses an electroless plating process applied on the surface of particles of nanoscale core metal powders (such as magnesium, aluminum, zinc, manganese and alloys thereof) to coat multiple layers of a nano-shell of different metals or metal oxides, such as Al, Ni, Al<sub>2</sub>O<sub>3</sub>, and the like, and then the composite powder is sintered to form a nanocomposite material having certain decomposition feature [sic].” Id.</p> <p>A POSITA in April 2014 would have understood that this sintering process discussed within Xiao was a well-known type of surface treatment that can be used to improve the surface hardness of the sintered metal. See also, e.g., Ex. 1203 (“Powder metal technology is well known to the persons skilled in the art and generally comprises the formation of metal powders which are compacted and then subjected to an elevated temperature so as to produce a sintered product.”). Consequently, Xiao teaches a magnesium composite subjected to a heat treating surface treatment to improve surface hardness of the magnesium.</p>
said surface treatment including peening, heat treatment, aluminizing, or combinations thereof.	As set forth in Ground I, supra, Xiao already discloses each and every element of the magnesium composition of Claim 1. Xiao does not claim, nor disclose in any novel embodiment, subjecting any of its novel magnesium composites to surface treatments (i.e. peening, heat treatment, aluminizing or combinations thereof) nor does it teach that surface treatment

	<p>would achieve any of the benefits set forth in Claim 8 of the ‘653 Patent.</p> <p>However, Xiao discloses that a U.S. Patent Publication teaches a method “for preparing a composite material having a decomposable multilayer coated core-shell structure (U.S. Patent No. US2011/0132143A1, 2011). ¶ 0002. It is explicitly noted in Xiao that this U.S. patent publication discloses an electroless plating process applied on the surface of particles of nanoscale core metal powders (such as magnesium, aluminum, zinc, manganese and alloys thereof) to coat multiple layers of a nano-shell of different metals or metal oxides, such as Al, Ni, Al<sub>2</sub>O<sub>3</sub>, and the like, and then the composite powder is sintered to form a nanocomposite material having certain decomposition feature [sic].” Id.</p> <p>A POSITA in April 2014 would have understood that this sintering process discussed within Xiao was a well-known type of surface treatment that can be used to improve the surface hardness of the sintered metal. See also, e.g., Ex. 1203 (“Powder metal technology is well known to the persons skilled in the art and generally comprises the formation of metal powders which are compacted and then subjected to an elevated temperature so as to produce a sintered product.”). Consequently, Xiao teaches a magnesium composite subjected to a heat treating surface treatment to improve surface hardness of the magnesium.</p>
<b>Independent Claim 25</b>	<p style="text-align: center;"><b>Disclosure of Claim Element in Xiao and/or Prior Art</b></p> <p style="text-align: center;"><b>Magnesium Disclosed in Xiao</b></p>
25. A dissolvable magnesium alloy composite for use in a ball or other tool component	Xiao discloses that the cast magnesium alloy “can be used as a tripping ball material for a multi-stage sliding sleeve staged-fracturing technique.” ¶ 0001. A POSITA would have

in a well drilling or completion operation,	understood that a tripping ball was a type of tool component in an oil well drilling operation. Medlin Decl. Exhibit 2 at ¶ 148.
said dissolvable magnesium alloy composite comprising at least 85 wt % magnesium;	<p>Claim 1 of Xiao discloses a “magnesium alloy, comprising the components at the weight percentages as follows: Al: 13 to 25%, Zn: 2 to 15%, the remainder is Mg, and a sum of the weight percentages of the components is 100%.” Xiao (emphasis added). Dr. Medlin opines that a POSITA would have understood that the range of non-magnesium elements in Claim 1 leaves 60-85 wt% for magnesium. Medlin Decl. Exhibit 2 at ¶ 149. Thus, Xiao discloses a range of magnesium up to 85 wt%, which touches the claimed range (i.e. at least 85 wt% magnesium) set forth in this claim element. However, this leaves no room for any of the claimed additive materials.</p> <p>Xiao also discloses a prior art magnesium alloy represented by Mg-Al-Zn (with AZ91D magnesium alloy being the most widely used) wherein the “main components of this alloy and the respective weight percentages are as follows: Al 8.3 to 9.7, Zn 0.35 to 1.0, Mn 0.15 to 0.5, and the remainder [(i.e. 88.8 to 91.2 wt%)] is magnesium.” Id. at ¶ 0003; Medlin Decl. Exhibit 2 at ¶ 269. Xiao also uses an AZ91D as a comparative example alloy, with a composition of 9 wt% Al, 1 wt% Zn, 0.3 wt% Zr, and 0.1 wt% Mn, with the remainder (i.e. 89.6 wt%) being magnesium. Id. at ¶ 0032. Xiao teaches a primary difference between this prior art and its novel magnesium alloy lies in the addition of metallic elements like Iron, Copper, and/or Nickel into the alloy. Id. at ¶ 0026 (“[e]lements such as Fe, Cu, Ni, Ag, etc., in the magnesium alloy can form a large number of intermetallic composite micro-particles, which can improve the corrosion performance of the magnesium alloy, thereby promoting the decomposition of the magnesium alloy”). The dissolution data in Xiao (Xiao at ¶ 0061) teaches that the</p>

	<p>addition of Iron, Copper, and/or Nickel (alongside the Aluminum, Zinc, Zirconium, and Manganese of the prior art AZ91D example) caused a surprisingly large increase in dissolution rates.</p> <p>Thus, Xiao, as a whole, teaches magnesium compositions with magnesium content above 85 wt% and “room” for the novel additive materials (i.e. “Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%” (Xiao at Claim 2)). Thus, Xiao fully discloses a range of magnesium of at least 85 wt %, touching and even overlapping the claimed range.</p>
one or more metals selected from the group consisting of 0.5-10 wt % aluminum, 0.05-6 wt % zinc, 0.01-3 wt % zirconium, and 0.15-2 wt % manganese; and about 0.05-45 wt % of a secondary metal to form a galvanically-active intermetallic particle that promotes corrosion of said dissolvable magnesium alloy composite,	<p>This claim element would be anticipated solely by the zirconium concentrations falling within the 0.01-3 wt% range. Claim 25 also requires, in addition to a similar zirconium concentration to the Markush claims, a zinc concentration of 0.05-6 wt%.</p> <p>Claim 1 of Xiao discloses “Al: 13 to 25%, Zn: 2 to 15%,” and Claim 2 of Xiao discloses secondary metals of “Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%; and a sum of the weight percentages of the components is 100%.” Thus, Xiao teaches 0.05-0.5 wt% zirconium, which touches and covers at least a portion of these claimed ranges. Medlin Decl. Exhibit 2 ¶ 150. Xiao similarly teaches 2-15 wt% zinc, which covers much of the claimed (0.05-6 wt%) zinc concentration. Id. at ¶¶ 167-68. A claim is invalid as anticipated when it is in Markush form and a prior art reference disclosed one of the claimed elements. See Fresenius USA, Inc. v. Baxter Int'l, Inc., 582 F.3d 1288, 1298 (Fed. Cir. 2009). See also Abbott Labs. v. Baxter Pharm. Prods., Inc., 334 F.3d 1274, 1280 (Fed.Cir.2003); Schering Corp. v. Geneva Pharms., Inc., 339 F.3d 1373, 1380 (Fed.Cir.2003);</p>

	<p>These metallic elements are found in the additive materials generally disclosed by Xiao (i.e., intermediate alloys: Al-Fe, Al-Ni, Al-Cu, Al-Ti) (Xiao at ¶ 0023) and particularly described in association with Example 7 of Xiao (Id. at ¶¶ 0057–0060) for which Dr. Medlin calculated for Example 7 of Xiao (Medlin Decl. Exhibit 2 at ¶¶ 113–122) the specific weight percentage (ranges) of each additive material or secondary metal. In particular, Dr. Medlin determined that the additive materials aluminum-iron with 30 wt% iron, aluminum-nickel with 45 wt% nickel, and aluminum-copper with 40 wt% copper also fall well within the range of about 0.05 wt% to 45 wt% of the mixture. Id. ¶ 122. For example, the aluminum-iron additive constitutes 3.3 wt% of the mixture; the aluminum-nickel additive constitutes 1.1 wt% of the mixture; and the aluminum-copper additive constitutes 0.25 wt% of the mixture. Dr. Medlin expressed his opinion that a POSITA in April 2014 would have also recognized that other intermediate alloy compositions would have fallen within the teachings of Example 7 and other disclosures of Xiao resulting in Al-Fe, Al-Ni, and Al-Cu intermediate alloys constituting about 0.05 wt% to 45 wt% of the mixture. Id. at ¶ 125. Accordingly, this claim element was disclosed by Xiao.</p>
said secondary metal including one or more metals selected from the group consisting of copper, nickel, cobalt, titanium and iron,	Xiao teaches the use of a variety of metals as additive materials. See, e.g., Xiao at ¶ 0026. For instance, Xiao claims that the novel magnesium alloy may “further compris[e] trace elements at the weight percentages as follows: Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%...”Id. at Claim 2. In addition, Example 7 of Xiao particularly discloses intermediate alloys Al-Fe, Al-Ni, Al-Ag, Al-Cu, Al-Zr, Al-Ti and Zinc as additive materials. Id. at ¶ 0060. A POSITA in April 2014 would have understood that all of these intermediate

	<p>alloys contain metal, including, in particular, copper, nickel, and iron, among other metals. Medlin Decl. Exhibit 2 at ¶ 145. Thus, Xiao discloses a number of metal additives, including copper, nickel, and iron and, hence fully discloses all of the limitations of these claim elements.</p>
said magnesium alloy composite has a dissolution rate of at least 5 mg/cm <sup>2</sup> /hr. in 3 wt % KCl water mixture at 90°C.	<p>A POSITA in April 2014 would have recognized that Xiao disclosed a table of dissolution rates (“decomposition rates”) because the units of measurement are the units used by POSITAs to measure dissolution rates. Medlin Decl. Exhibit 2 at ¶ 129. The disclosed table provides seven examples (excluding the comparative [prior art] example). Xiao at ¶ 0064. Xiao provides dissolution rates in g/cm<sup>2</sup>/hr, while the claims of the ’653 Patent express dissolution rates in mg/cm<sup>2</sup>/hr. A POSITA would have known converting between g/cm<sup>2</sup>/hr to mg/cm<sup>2</sup>/hr, merely requires multiplication by 1,000. Medlin Decl. Exhibit 2 at ¶ 130.</p> <p>Thus, each of Xiao’s seven examples (excluding the comparative [prior art] example) has a dissolution rate of at least three times the claimed “at least 5 mg/cm<sup>2</sup>/hr in 3 wt.% KCl water” at both 70°C or 93°C (with Example 7 being more than seven times that floor), Dr. Medlin opines that a POSITA in April 2014 would have recognized that the dissolution rate of the examples in Xiao would necessarily have been well above the claimed floor of “at least of 5 mg/cm<sup>2</sup>/hr” at the claimed 90°C. Id. at ¶ 132.</p>
<b>Dependent Claim 26</b>	<p style="text-align: center;"><b>Disclosure of Claim Element in Xiao and/or Prior Art</b></p> <p style="text-align: center;"><b>Magnesium Disclosed in Xiao</b></p>
26. The dissolvable magnesium alloy composite as defined in claim 25, wherein a dissolution rate of	As the discussion, supra, demonstrates, the novel examples explicitly analyzed in Xiao disclose dissolution rates only up to 74 mg/cm <sup>2</sup> /hr in 3 wt.% KCl water at 93°C, shy of this claimed

said magnesium alloy composite is 100-325 mg/cm <sup>2</sup> /hr. in 3 wt % KCl water mixture at 90°C.	<p>range. Medlin Decl. Exhibit 2 at ¶ 330. However, Dr. Medlin opines that a POSITA in April 2014 would have understood Xiao to also teach dissolution rates above that floor at 90°C, because (1) the dissolution rates in the Xiao examples are very close to the claimed floor (e.g. Example 1 exhibited a rate of 74 mg/cm<sup>2</sup>/hr at 93°C, Example 6 a rate of 63 mg/cm<sup>2</sup>/hr at 90°C, and Example 7 a rate of 57 mg/cm<sup>2</sup>/hr at 93°C; and (2) a POSITA would have expected that other magnesium compositions having additive amounts in the ranges as disclosed by Xiao, but with higher amounts of copper, nickel, and/or iron than the amounts tested by Xiao's examples would have improved corrosion performance. Medlin Decl. Exhibit 2 at ¶ 331. In particular, even though they come close to the 100 mg/cm<sup>2</sup>/hr at 90°C, Examples 1, 6 and 7 of Xiao had less aggregate copper, nickel and iron than taught by Claim 2 of Xiao (i.e. 5 wt% for each of copper, nickel, and iron in a magnesium composite). Medlin Decl. Exhibit 2 at ¶ 331. Thus, the Examples in Xiao used lower amounts of these specific dissolution-enhancing additive elements. Thus, according to Dr. Medlin, a POSITA in April 2014 would have understood this and recognized that Xiao teaches magnesium compositions having dissolution rates above the claimed floor.</p>
	<p>Dr. Medlin also notes that it was well-known prior to April 2014 that additions of copper, iron, nickel and cobalt cause a sharp linear increase in the corrosion rate of magnesium as evidenced by Shaw, "Corrosion Resistance of Magnesium Alloys" published in 2003 in the ASM Handbook, Vol. 13A (2003). Ex. 1204. Medlin Decl. Exhibit 2 at ¶ 332. As such, the recitation of the claimed dissolution rate of 100-325 mg/cm<sup>2</sup>/hr at 90°C was not a new property or use but rather a property that would</p>

	be inherent in the compositions disclosed in Xiao, as confirmed by the extrinsic evidence of Shaw.
<b>Dependent Claim 27</b>	<b>Disclosure of Claim Element in Xiao and/or Prior Art</b> <b>Magnesium Disclosed in Xiao</b>
27. The dissolvable magnesium composite as defined in claim 25, wherein said secondary metal includes nickel,	Xiao discloses a “magnesium alloy...and further adds elements of Fe, Cu, Ni, and Ag which can enhance the corrosion performance of the magnesium alloy...” Xiao at ¶ 0026, ll. 1–9. See Declaration of Dr. Dana Medlin (Medlin Decl. Exhibit 2, e.g., at ¶ 104-105 (Claim 1)).  Xiao teaches the use of a variety of metals as additive materials. See, e.g., Xiao at ¶ 0026. For instance, Xiao claims that the novel magnesium alloy may “further compris[e] trace elements at the weight percentages as follows: Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%...”Id. at Claim 2. In addition, Example 7 of Xiao particularly discloses intermediate alloys Al-Fe, Al-Ni, Al-Ag, Al-Cu, Al-Zr, Al-Ti and Zinc as additive materials. Id. at ¶ 0060. A POSITA in April 2014 would have understood that all of these intermediate alloys contain metal, including nickel, among other metals. Medlin Decl. Exhibit 2 at ¶ 145. Thus, Xiao discloses a number of metal additives, including nickel, and hence fully discloses all of the limitations of these claim elements.
said nickel forms galvanically-active in situ precipitate in said magnesium composite.	Xiao discloses a novel magnesium alloy with high aluminum and zinc content further adding elements Fe, Cu, Ni and Ag which can form intermetallics and enhance the corrosion performance of the magnesium alloy. Xiao at ¶ 0026. Xiao explains the corrosion mechanism within a magnesium alloy in terms of a cathode phase, which Xiao explains includes “the $\beta$ ( $Mg_{17}Al_{12}$ ) phase and eutectic phase on the grain boundary of the magnesium alloy,” and “the magnesium matrix $\alpha$ phase which functions as an anode phase, so that the matrix and the

	<p>grain boundary of the magnesium alloy form a large amount of micro-batteries, which [micro-batteries] greatly accelerate the corrosion decomposition of magnesium alloy.” Id.</p> <p>These elements are among the claimed “secondary metals” disclosed by Xiao. Thus, all of the limitations of this claim element are disclosed by Xiao.</p>
<b>Dependent Claim 28</b>	<p style="text-align: center;"><b>Disclosure of Claim Element in Xiao and/or Prior Art</b></p> <p style="text-align: center;"><b>Magnesium Disclosed in Xiao</b></p>
28. The dissolvable magnesium alloy composite as defined in claim 25, wherein said additive material is a metal or metal alloy.	<p>Xiao discloses a “magnesium alloy...and further adds elements of Fe, Cu, Ni, and Ag which can enhance the corrosion performance of the magnesium alloy...” Xiao at ¶ 0026, ll. 1–9. See Declaration of Dr. Dana Medlin (Medlin Decl. Exhibit 2, e.g., at ¶ 104-105 (Claim 1)).</p> <p>Xiao teaches the use of a variety of metals as additive materials. See, e.g., Xiao at ¶ 0026. For instance, Xiao claims that the magnesium alloy may “further compris[e] trace elements at the weight percentages as follows: Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%...” Id. at Claim 2. In addition, Example 7 of Xiao particularly discloses intermediate alloys Al-Fe, Al-Ni, Al-Ag, Al-Cu, Al-Zr, Al-Ti and Zinc as additive materials. Id. at ¶ 0060. A POSITA in April 2014 would have understood that all of these additive materials contain metal(s). Medlin Decl. Exhibit 2 at ¶ 145. Accordingly, Example 7 of Xiao discloses this claim element in accordance with Patent Owner’s definition of “additive material.”</p>
<b>Independent Claim 33</b>	<p style="text-align: center;"><b>Disclosure of Claim Element in Xiao and/or Prior Art</b></p> <p style="text-align: center;"><b>Magnesium Disclosed in Xiao</b></p>
33. A dissolvable magnesium alloy composite for use in a ball or other tool component	Xiao discloses a “magnesium alloy...and further adds elements of Fe, Cu, Ni, and Ag which can enhance the corrosion performance of the magnesium alloy...” Xiao at ¶ 0026, ll. 1–9.

in a well drilling or completion operation,	<p>See Declaration of Dr. Dana Medlin (Medlin Decl. Exhibit 2, e.g., at ¶¶ 104-105 (Claim 1)).</p> <p>Xiao discloses that the cast magnesium alloy “can be used as a tripping ball material for a multi-stage sliding sleeve staged-fracturing technique.” ¶ 0001. A POSITA would have understood that a tripping ball was a type of tool component in an oil well drilling operation. Medlin Decl. Exhibit 2 at ¶ 148.</p>
said dissolvable magnesium alloy composite comprising 60-95 wt % magnesium; 0.5-10 wt % aluminum; 0.05-6 wt % zinc; 0.15-2 wt % manganese; and about 0.05-45 wt % of a secondary metal	<p>Claim 1 of Xiao discloses a “magnesium alloy, comprising the components at the weight percentages as follows: Al: 13 to 25%, Zn: 2 to 15%; the remainder is Mg, and a sum of the weight percentages of the components is 100%.” Xiao at Claim 1 (emphasis added). Dr. Medlin opines that a POSITA would have understood that the total range of the non-magnesium elements disclosed in Claim 1 of Xiao is 15 to 40%, leaving 60-85 wt% for magnesium. Medlin Decl. Exhibit 2 at ¶ 149. Dr. Medlin also notes that the “Examples of Xiao disclose a range of magnesium from 54.5 wt% - 79.2 wt%.” Id. Thus, Xiao discloses a range of magnesium 54.5-85 wt%, which overlaps and covers much of the claimed ranges set forth in these claim elements.</p> <p>Claim 2 discloses: a “magnesium alloy, according to Claim 1, further comprising the trace elements at the weight percentages as follows: Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%; and a sum of the weight percentages of the components is 100%.” Id. at Claim 2.</p> <p>These metallic elements are found in the additive materials generally disclosed by Xiao (i.e., intermediate alloys: Al-Fe, Al-Ni, Al-Cu, Al-Ti) (Xiao at ¶ 0023) and particularly described in association with Example 7 of Xiao (Id. at ¶¶ 0057-0060) for which Dr. Medlin calculated for Example 7 of Xiao (Medlin Decl. Exhibit 2 at ¶¶ 113-122) the specific weight percentage</p>

(ranges) of each additive material or secondary metal. In particular, Dr. Medlin determined that the additive materials aluminum-iron with 30 wt% iron, aluminum-nickel with 45 wt% nickel, and aluminum-copper with 40 wt% copper also fall well within the range of about 0.05 wt% to 45 wt% of the mixture. Id. ¶ 122. For example, the aluminum-iron additive constitutes 3.3 wt% of the mixture; the aluminum-nickel additive constitutes 1.1 wt% of the mixture; and the aluminum-copper additive constitutes 0.25 wt% of the mixture. Dr. Medlin expressed his opinion that a POSITA in April 2014 would have also recognized that other intermediate alloy compositions would have fallen within the teachings of Example 7 and other disclosures of Xiao resulting in Al-Fe, Al-Ni, and Al-Cu intermediate alloys constituting about 0.05 wt% to 45 wt% of the mixture. Id. at ¶ 125. Accordingly, these claim elements are disclosed by Xiao.

Claim 1 of Xiao discloses a “magnesium alloy, comprising the components at the weight percentages as follows: Al: 13 to 25%, Zn: 2 to 15%, the remainder is Mg, and a sum of the weight percentages of the components is 100%.” Xiao (emphasis added). Dr. Medlin opines that a POSITA would have understood that the range of non-magnesium elements in Claim 1 leaves 60-85 wt% for magnesium. Medlin Decl. Exhibit 2 at ¶ 149. Thus, Xiao discloses a range of magnesium up to 85 wt%, which touches the claimed range (i.e. at least 85 wt% magnesium) set forth in these claim elements. However, this leaves no room for any of the claimed additive materials. Xiao also discloses a prior art magnesium alloy represented by Mg-Al-Zn (with AZ91D magnesium alloy being the most widely used) wherein the “main components of this alloy and

	<p>the respective weight percentages are as follows: Al 8.3 to 9.7, Zn 0.35 to 1.0, Mn 0.15 to 0.5, and the remainder [(i.e. 88.8 to 91.2 wt%)] is magnesium.” Id. at ¶ 0003; Medlin Decl. Exhibit 2 at ¶ 269. Xiao also uses an AZ91D as a comparative example alloy, with a composition of 9 wt% Al, 1 wt% Zn, 0.3 wt% Zr, and 0.1 wt% Mn, with the remainder (i.e. 89.6 wt%) being magnesium. Id. at ¶ 0032. Xiao teaches a primary difference between this prior art and its novel magnesium alloy lies in the addition of metallic elements like Iron, Copper, and/or Nickel into the alloy. Id. at ¶ 0026 (“[e]lements such as Fe, Cu, Ni, Ag, etc., in the magnesium alloy can form a large number of intermetallic composite micro-particles, which can improve the corrosion performance of the magnesium alloy, thereby promoting the decomposition of the magnesium alloy”). The dissolution data in Xiao (Xiao at ¶ 0061) teaches that the addition of Iron, Copper, and/or Nickel (alongside the Aluminum, Zinc, Zirconium, and Manganese of the prior art AZ91D example) caused a surprisingly large increase in dissolution rates.</p> <p>Thus, Xiao, as a whole, teaches magnesium compositions with magnesium content above 85wt% and “room” for the novel additive materials (i.e. “Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%” (Xiao at Claim 2)). Thus, Xiao fully discloses a range of magnesium of at least 85 wt %, touching and even overlapping the claim range.</p>
to form a galvanically-active intermetallic particle that promotes corrosion of said dissolvable magnesium alloy composite,	Xiao discloses a novel magnesium alloy with high aluminum and zinc content further adding elements Fe, Cu, Ni and Ag which can form intermetallics and enhance the corrosion performance of the magnesium alloy. Xiao at ¶ 0026. Xiao explains the corrosion mechanism within a magnesium alloy in

	<p>terms of a cathode phase, which Xiao explains includes “the <math>\beta</math> (<math>Mg_{17}Al_{12}</math>) phase and eutectic phase on the grain boundary of the magnesium alloy,” and “the magnesium matrix <math>\alpha</math> phase which functions as an anode phase, so that the matrix and the grain boundary of the magnesium alloy form a large amount of micro-batteries, which [micro- batteries] greatly accelerate the corrosion decomposition of magnesium alloy.” Id.</p> <p>A POSITA in August 2014 would have understood this disclosure of “intermetallic composite micro-particles” to be a reference to intermetallic precipitate. Medlin Decl. Exhibit 2 at ¶ 107.</p> <p>A POSITA reading the Xiao reference in April 2014 would have recognized that the precipitation of galvanically-active intermetallic phases (“micro-batteries”) are naturally and necessarily generated by the in situ reaction between magnesium and any and all of the disclosed metal additives forming in situ precipitate. Medlin Decl. Exhibit 2, at ¶ 108. Thus, these claim elements were disclosed by Xiao.</p> <p>These elements are among the claimed “secondary metals” disclosed by Xiao. Thus, all of the limitations of this claim element are disclosed by Xiao.</p>
said secondary metal including one or more metals selected from the group consisting of copper, nickel, cobalt, titanium and iron,	Xiao teaches the use of a variety of metals as additive materials. See, e.g., Xiao at ¶ 0026. For instance, Xiao claims that the novel magnesium alloy may “further compris[e] trace elements at the weight percentages as follows: Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%...” Id. at Claim 2. In addition, Example 7 of Xiao particularly discloses intermediate alloys Al-Fe, Al-Ni, Al-Ag, Al-Cu, Al-Zr, Al-Ti and Zinc as additive materials. Id. at ¶ 0060. A POSITA in April 2014 would have understood that all of these intermediate alloys contain metal, including, in particular, copper, nickel,

	and iron, among other metals. Medlin Decl. Exhibit 2 at ¶ 145. Thus, Xiao discloses a number of metal additives, including copper, nickel, and iron and, hence fully discloses all of the limitations of these claim elements.
said magnesium alloy composite has a dissolution rate of at least 5 mg/cm <sup>2</sup> /hr. in 3 wt % KCl water mixture at 90°C.	A POSITA in April 2014 would have recognized that Xiao disclosed a table of dissolution rates (“decomposition rates”) because the units of measurement are the units used by POSITAs to measure dissolution rates. Medlin Decl. Exhibit 2 at ¶ 129. The disclosed table provides seven examples (excluding the comparative [prior art] example). Xiao at ¶ 0064. Xiao provides dissolution rates in g/cm <sup>2</sup> /hr, while the claims of the ’653 Patent express dissolution rates in mg/cm <sup>2</sup> /hr. A POSITA would have known converting between g/cm <sup>2</sup> /hr to mg/cm <sup>2</sup> /hr, merely requires multiplication by 1,000. Medlin Decl. Exhibit 2 at ¶ 130.  Thus, each of Xiao’s seven examples (excluding the comparative [prior art] example) has a dissolution rate of at least three times the claimed “at least 5 mg/cm <sup>2</sup> /hr in 3 wt.% KCl water” at both 70°C or 93°C (with Example 7 being more than seven times that floor), Dr. Medlin opines that a POSITA in April 2014 would have recognized that the dissolution rate of the examples in Xiao would necessarily have been well above the claimed floor of “at least of 5 mg/cm <sup>2</sup> /hr” at the claimed 90°C. Id. at ¶ 132.
<b>Dependent Claim 34</b>	<b>Disclosure of Claim Element in Xiao and/or Prior Art</b> <b>Magnesium Disclosed in Xiao</b>
34. The dissolvable magnesium alloy composite as defined in claim 33, wherein a dissolution rate of said magnesium alloy	A POSITA in April 2014 would have recognized that Xiao disclosed a table of dissolution rates (“decomposition rates”) because the units of measurement are the units used by POSITAs to measure dissolution rates. Medlin Decl. Exhibit 2

composite is 5-325 mg/cm <sup>2</sup> /hr. in 3 wt % KCl water mixture at 90°C.	<p>at ¶ 129. The disclosed table provides seven examples (excluding the comparative [prior art] example). Xiao at ¶ 0064. Xiao provides dissolution rates in g/cm<sup>2</sup>/hr, while the claims of the '653 Patent express dissolution rates in mg/cm<sup>2</sup>/hr. A POSITA would have known converting between g/cm<sup>2</sup>/hr to mg/cm<sup>2</sup>/hr, merely requires multiplication by 1,000. Medlin Decl. Exhibit 2 at ¶ 130.</p> <p>Thus, each of Xiao's seven examples (excluding the comparative [prior art] example) has a dissolution rate within the claimed range of at least three times the claimed "about 5-325 mg/cm<sup>2</sup>/hr in 3 wt.% KCl water" at both 70°C or 93°C (with Example 7 being more than seven times that floor), Dr. Medlin opines that a POSITA in April 2014 would have recognized that the dissolution rate of the examples in Xiao would necessarily have been well above the claimed floor of "about 5" "about 5-325 mg/cm<sup>2</sup>/hr" at the claimed 90°C. Id. at ¶ 132.</p>
<b>Dependent Claim 35</b>	<b>Disclosure of Claim Element in Xiao and/or Prior Art</b> <b>Magnesium Disclosed in Xiao</b>
35. The dissolvable magnesium composite as defined in claim 33, wherein said secondary metal includes nickel,	<p>Xiao discloses a "magnesium alloy...and further adds elements of Fe, Cu, Ni, and Ag which can enhance the corrosion performance of the magnesium alloy..." Xiao at ¶ 0026, ll. 1-9. See Declaration of Dr. Dana Medlin (Medlin Decl. Exhibit 2, e.g., at ¶ 104-105 (Claim 1)).</p> <p>Xiao teaches the use of a variety of metals as additive materials. See, e.g., Xiao at ¶ 0026. For instance, Xiao claims that the novel magnesium alloy may "further compris[e] trace elements at the weight percentages as follows: Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%..."Id. at Claim 2. In addition, Example 7 of Xiao particularly discloses intermediate alloys Al-Fe, Al-Ni, Al-Ag, Al-Cu, Al-Zr, Al-Ti</p>

	and Zinc as additive materials. Id. at ¶ 0060. A POSITA in April 2014 would have understood that all of these intermediate alloys contain metal, including nickel, among other metals. Medlin Decl. Exhibit 2 at ¶ 145. Thus, Xiao discloses a number of metal additives, including nickel, and hence fully discloses all of the limitations of these claim elements.
said nickel forms galvanically-active in situ precipitate in said magnesium composite.	Xiao discloses a novel magnesium alloy with high aluminum and zinc content further adding elements Fe, Cu, Ni and Ag which can form intermetallics and enhance the corrosion performance of the magnesium alloy. Xiao at ¶ 0026. Xiao explains the corrosion mechanism within a magnesium alloy in terms of a cathode phase, which Xiao explains includes “the $\beta$ ( $Mg_{17}Al_{12}$ ) phase and eutectic phase on the grain boundary of the magnesium alloy,” and “the magnesium matrix $\alpha$ phase which functions as an anode phase, so that the matrix and the grain boundary of the magnesium alloy form a large amount of micro-batteries, which [micro-batteries] greatly accelerate the corrosion decomposition of magnesium alloy.” Id.  A POSITA in August 2014 would have understood this disclosure of “intermetallic composite micro-particles” to be a reference to intermetallic precipitate. Medlin Decl. Exhibit 2 at ¶ 107.  A POSITA reading the Xiao reference in April 2014 would have recognized that the precipitation of galvanically-active intermetallic phases (“micro-batteries”) are naturally and necessarily generated by the in situ reaction between magnesium and any and all of the disclosed metal additives forming in situ precipitate. Medlin Decl. Exhibit 2, at ¶ 108. Thus, these claim elements were disclosed by Xiao.

	These elements are among the claimed “secondary metals” disclosed by Xiao. Thus, all of the limitations of this claim element are disclosed by Xiao.
<b>Dependent Claim 36</b>	<b>Disclosure of Claim Element in Xiao and/or Prior Art</b> <b>Magnesium Disclosed in Xiao</b>
36. The dissolvable magnesium alloy composite as defined in claim 33, wherein said additive material is a metal or metal alloy.	Xiao discloses a “magnesium alloy...and further adds elements of Fe, Cu, Ni, and Ag which can enhance the corrosion performance of the magnesium alloy...” Xiao at ¶ 0026, ll. 1–9. See Declaration of Dr. Dana Medlin (Medlin Decl. Exhibit 2, e.g., at ¶ 104-105 (Claim 1)).  Xiao teaches the use of a variety of metals as additive materials. See, e.g., Xiao at ¶ 0026. For instance, Xiao claims that the magnesium alloy may “further compris[e] trace elements at the weight percentages as follows: Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%. . .” Id. at Claim 2. In addition, Example 7 of Xiao particularly discloses intermediate alloys Al-Fe, Al-Ni, Al-Ag, Al-Cu, Al-Zr, Al-Ti and Zinc as additive materials. Id. at ¶ 0060. A POSITA in April 2014 would have understood that all of these additive materials contain metal(s). Medlin Decl. Exhibit 2 at ¶ 145. Accordingly, Example 7 of Xiao discloses this claim element in accordance with Patent Owner’s definition of “additive material.”
<b>Dependent Claim 55</b>	<b>Disclosure of Claim Element in Xiao and/or Prior Art</b> <b>Magnesium Disclosed in Xiao</b>
55. The magnesium composite as defined in claim 49, wherein said magnesium alloy includes at least 85 wt % magnesium, and one or	Xiao discloses a “magnesium alloy...and further adds elements of Fe, Cu, Ni, and Ag which can enhance the corrosion performance of the magnesium alloy...” Xiao at ¶ 0026, ll. 1–9. See Declaration of Dr. Dana Medlin (Medlin Decl. Exhibit 2, e.g., at ¶ 104-105 (Claim 1)).

<p>more metals selected from the group consisting of 0.5-10 wt % aluminum, 0.05-6 wt % zinc, 0.01-3 wt % zirconium, and 0.15-2 wt % manganese.</p>	<p>This claim element would be anticipated solely by the zirconium concentrations falling within the 0.01-3 wt% range. Claim 55 also requires, in addition to a similar zirconium concentration to the Markush claims, a zinc concentration of 0.05-6 wt%.</p> <p>Claim 1 of Xiao discloses “Al: 13 to 25%, Zn: 2 to 15%,” and Claim 2 of Xiao discloses secondary metals of “Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%; and a sum of the weight percentages of the components is 100%.” Thus, Xiao teaches 0.05-0.5 wt% zirconium, which touches and covers at least a portion of these claimed ranges. Medlin Decl. Exhibit 2 ¶ 150. Xiao similarly teaches 2-15 wt% zinc, which covers much of the claimed (0.05-6 wt%) zinc concentration. Id. at ¶¶ 167-68. A claim is invalid as anticipated when it is in Markush form and a prior art reference disclosed one of the claimed elements. See Fresenius USA, Inc. v. Baxter Int'l, Inc., 582 F.3d 1288, 1298 (Fed. Cir. 2009). See also Abbott Labs. v. Baxter Pharm. Prods., Inc., 334 F.3d 1274, 1280 (Fed.Cir.2003); Schering Corp. v. Geneva Pharms., Inc., 339 F.3d 1373, 1380 (Fed.Cir.2003); Thus, Xiao teaches all of these claim limitations.</p> <p>Claim 1 of Xiao discloses a “magnesium alloy, comprising the components at the weight percentages as follows: Al: 13 to 25%, Zn: 2 to 15%, the remainder is Mg, and a sum of the weight percentages of the components is 100%.” Xiao (emphasis added). Dr. Medlin opines that a POSITA would have understood that the range of non-magnesium elements in Claim 1 leaves 60-85 wt% for magnesium. Medlin Decl. Exhibit 2 at ¶ 149. Thus, Xiao discloses a range of magnesium up to 85 wt%, which touches the claimed range (i.e. at least 85</p>
--	--

	<p>wt% magnesium) set forth in these claim elements. However, this leaves no room for any of the claimed additive materials. Xiao also discloses a prior art magnesium alloy represented by Mg-Al-Zn (with AZ91D magnesium alloy being the most widely used) wherein the “main components of this alloy and the respective weight percentages are as follows: Al 8.3 to 9.7, Zn 0.35 to 1.0, Mn 0.15 to 0.5, and the remainder [(i.e. 88.8 to 91.2 wt%)] is magnesium.” Id. at ¶ 0003; Medlin Decl. Exhibit 2 at ¶ 269. Xiao also uses an AZ91D as a comparative example alloy, with a composition of 9 wt% Al, 1 wt% Zn, 0.3 wt% Zr, and 0.1 wt% Mn, with the remainder (i.e. 89.6 wt%) being magnesium. Id. at ¶ 0032. Xiao teaches a primary difference between this prior art and its novel magnesium alloy lies in the addition of metallic elements like Iron, Copper, and/or Nickel into the alloy. Id. at ¶ 0026 (“[e]lements such as Fe, Cu, Ni, Ag, etc., in the magnesium alloy can form a large number of intermetallic composite micro-particles, which can improve the corrosion performance of the magnesium alloy, thereby promoting the decomposition of the magnesium alloy”). The dissolution data in Xiao (Xiao at ¶ 0061) teaches that the addition of Iron, Copper, and/or Nickel (alongside the Aluminum, Zinc, Zirconium, and Manganese of the prior art AZ91D example) caused a surprisingly large increase in dissolution rates.</p> <p>Thus, Xiao, as a whole, teaches magnesium compositions with magnesium content above 85wt% and “room” for the novel additive materials (i.e. “Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%” (Xiao at Claim 2)). Thus, Xiao fully discloses a range of magnesium of at least 85 wt %, touching and even overlapping the claim range.</p>
--	---

	As for the other requirements of dependent claim 55, it has already been established in Ground I that Xiao discloses each and every element of independent claim 49. Ground I also establishes that Xiao discloses at least three of the metals overlapping the weight percentage ranges required by the Markush element of dependent claim 55 (i.e., 0.5-10 wt% aluminum, 0.05-6wt% zinc, and/or 0.01-3 wt% zirconium).
<b>Independent Claim 74</b>	<b>Disclosure of Claim Element in Xiao and/or Prior Art</b> <b>Magnesium Disclosed in Xiao</b>
74. A dissolvable magnesium composite for use in a ball or other tool component in a well drilling or completion operation,	Xiao discloses a “magnesium alloy...and further adds elements of Fe, Cu, Ni, and Ag which can enhance the corrosion performance of the magnesium alloy...” Xiao at ¶ 0026, ll. 1–9. See Declaration of Dr. Dana Medlin (Medlin Decl. Exhibit 2, e.g., at ¶ 104-105 (Claim 1)).  Xiao discloses that the cast magnesium alloy “can be used as a tripping ball material for a multi-stage sliding sleeve staged-fracturing technique.” Xiao at ¶ 0001. A POSITA would have understood that a tripping ball was a type of tool component in an oil well drilling operation. Medlin Decl. Exhibit 2 at ¶ 148. Thus, these claim elements were disclosed by Xiao.
said dissolvable magnesium composite includes in situ precipitation of galvanically-active intermetallic phases to enable controlled dissolution of said magnesium composite,	Xiao discloses a novel magnesium alloy with high aluminum and zinc content further adding elements Fe, Cu, Ni and Ag which can form intermetallics and enhance the corrosion performance of the magnesium alloy. Xiao explains the corrosion mechanism within a magnesium alloy in terms of a cathode phase, which Xiao explains includes “the $\beta$ ( $Mg_{17}Al_{12}$ ) phase and eutectic phase on the grain boundary of the magnesium alloy,” and “the magnesium matrix $\alpha$ phase which functions as an anode phase, so that the matrix and the grain boundary of the magnesium alloy form a large amount of

	<p>micro-batteries, which [micro-batteries] greatly accelerate the corrosion decomposition of magnesium alloy.” Xiao at ¶ 0026. A POSITA in August 2014 would have understood this disclosure of “intermetallic composite micro-particles” to be a reference to intermetallic precipitate. Medlin Decl. Exhibit 2 at ¶ 107.</p> <p>A POSITA reading the Xiao reference in April 2014 would have recognized that the precipitation of galvanically-active intermetallic phases (“micro-batteries”) are naturally and necessarily generated by the <i>in situ</i> reaction between magnesium and any and all of the disclosed metal additives forming <i>in situ</i> precipitate. Medlin Decl. Exhibit 2, at ¶ 108. Thus, these claim elements were disclosed by Xiao.</p>
said magnesium composite comprising a mixture of magnesium or a magnesium alloy and an additive material,	Xiao discloses “first loading pure magnesium and pure aluminum into a smelting furnace . . . next loading pure zinc and an [sic] intermediate alloys of trace element components into a resulting magnesium-aluminum alloy melt after melting.” Xiao, at ¶ 0022. Xiao discloses that “Al-Fe . . . Al-Ni . . . Al-Cu . . . Al-Ag . . . Al-Zr . . . and . . . Al-Ti intermediate alloy[s], are heated to dry and then added to the magnesium-aluminum alloy melt.” Id. at ¶ 0023. These intermediate alloys meet the Patent Owner’s definition of “additive material,” i.e., “a material that is added.” Medlin Decl. Exhibit 2, at ¶ 110. The addition of these intermediate alloys to the melted magnesium alloy necessarily results in a mixture of the additive material and magnesium alloy. Id. at ¶ 111.
said additive material constituting at least 0.1 wt % of said mixture,	Claim 1 of Xiao discloses: a “magnesium alloy, comprising the components at the weight percentages as follows: Al: 13 to 25%, Zn: 2 to 15%, the remainder is Mg, and a sum of the weight percentages of the components is 100%.” Xiao at Claim 1. And Claim 2 discloses: a “magnesium alloy, according to

	<p>Claim 1, further comprising the trace elements at the weight percentages as follows: Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%; and a sum of the weight percentages of the components is 100%.” Id. at Claim 2.</p> <p>These metallic elements are found in the additive materials generally disclosed by Xiao (i.e., intermediate alloys: Al-Fe, Al-Ni, Al-Cu, Al-Ti) (Xiao at ¶ 0023) and particularly described in association with Example 7 of Xiao (Id. at ¶¶ 0057–0060) for which Dr. Medlin calculated for Example 7 of Xiao (Medlin Decl. Exhibit 2 at ¶¶ 113–122) the specific weight percentage (ranges) of each additive material or secondary metal. In particular, Dr. Medlin determined that the additive materials aluminum-iron with 30 wt% iron, aluminum-nickel with 45 wt% nickel, and aluminum-copper with 40 wt% copper also fall well within the range of about 0.05 wt% to 45 wt% of the mixture. Id. ¶ 122. For example, the aluminum-iron additive constitutes 3.3 wt% of the mixture; the aluminum-nickel additive constitutes 1.1 wt% of the mixture; and the aluminum-copper additive constitutes 0.25 wt% of the mixture. Dr. Medlin expressed his opinion that a POSITA in April 2014 would have also recognized that other intermediate alloy compositions would have fallen within the teachings of Example 7 and other disclosures of Xiao resulting in Al-Fe, Al-Ni, and Al-Cu intermediate alloys constituting about 0.05 wt% to 45 wt% of the mixture. Id. at ¶ 125. Accordingly, these claim elements are disclosed by Xiao</p>
said magnesium in said magnesium composite constituting at least 85 wt %,	Claim 1 of Xiao discloses a “magnesium alloy, comprising the components at the weight percentages as follows: Al: 13 to 25%, Zn: 2 to 15%, the remainder is Mg, and a sum of the weight percentages of the components is 100%.” Xiao

(emphasis added). Dr. Medlin opines that a POSITA would have understood that the range of non-magnesium elements in Claim 1 leaves 60-85 wt% for magnesium. Medlin Decl. Exhibit 2 at ¶ 149. Thus, Xiao discloses a range of magnesium up to 85 wt%, which overlaps with the claimed range (i.e. at least 85wt% magnesium means 85wt% or greater).

Xiao also discloses a prior art magnesium alloy represented by Mg-Al-Zn (with AZ91D magnesium alloy being the most widely used) wherein the “main components of this alloy and the respective weight percentages are as follows: Al 8.3 to 9.7, Zn 0.35 to 1.0, Mn 0.15 to 0.5, and the remainder [(i.e. 88.8 to 91.2 wt%)] is magnesium.” Id. at ¶ 0003; Medlin Decl. Exhibit 2 at ¶ 269. Xiao also uses an AZ91D as a comparative example alloy, with a composition of 9 wt% Al, 1 wt% Zn, 0.3 wt% Zr, and 0.1 wt% Mn, with the remainder (i.e. 89.6 wt%) being magnesium. Id. at ¶ 0032. Xiao teaches a primary difference between this prior art and its novel magnesium alloy lies in the addition of metallic elements like Iron, Copper, and/or Nickel into the alloy. Id. at ¶ 0026 (“[e]lements such as Fe, Cu, Ni, Ag, etc., in the magnesium alloy can form a large number of intermetallic composite micro-particles, which can improve the corrosion performance of the magnesium alloy, thereby promoting the decomposition of the magnesium alloy”). The dissolution data in Xiao (Xiao at ¶ 0061) teaches that the addition of Iron, Copper, and/or Nickel (alongside the Aluminum, Zinc, Zirconium, and Manganese of the prior art AZ91D example) caused a surprisingly large increase in dissolution rates.

Thus, Xiao, as a whole, teaches magnesium compositions with magnesium content above 85wt% and “room” for the novel

	<p>additive materials (i.e. “Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%” (Xiao at Claim 2)). Thus, Xiao fully discloses a range of magnesium of at least 85 wt %.</p>
said additive material is a metal material selected from the group consisting of copper, nickel and cobalt,	<p>Xiao teaches the use of a variety of metals as additive materials. See, e.g., Xiao at ¶ 0026. For instance, Xiao claims that the novel magnesium alloy may “further compris[e] trace elements at the weight percentages as follows: Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%...”Id. at Claim 2. In addition, Example 7 of Xiao particularly discloses intermediate alloys Al-Fe, Al-Ni, Al-Ag, Al-Cu, Al-Zr, Al-Ti and Zinc as additive materials. Id. at ¶ 0060. A POSITA in April 2014 would have understood that all of these intermediate alloys contain metal, including, in particular, copper, nickel, and iron, among other metals. Medlin Decl. Exhibit 2 at ¶ 145. Thus, Xiao discloses a number of metal additives, including copper, nickel, and iron and, hence fully discloses all of the limitations of these claim elements.</p>
said magnesium composite including in situ precipitation of galvanically-active intermetallic phases that include said additive material,	<p>Xiao discloses a novel magnesium alloy with high aluminum and zinc content further adding elements Fe, Cu, Ni and Ag which can form intermetallics and enhance the corrosion performance of the magnesium alloy. Xiao at ¶ 0026. Xiao explains the corrosion mechanism within a magnesium alloy in terms of a cathode phase, which Xiao explains includes “the <math>\beta</math> (<math>Mg_{17}Al_{12}</math>) phase and eutectic phase on the grain boundary of the magnesium alloy,” and “the magnesium matrix <math>\alpha</math> phase which functions as an anode phase, so that the matrix and the grain boundary of the magnesium alloy form a large amount of micro-batteries, which [micro- batteries] greatly accelerate the corrosion decomposition of magnesium alloy.” Id.</p>

	<p>As Dr. Medlin explains, a POSITA in August 2014 would have understood this disclosure of “intermetallic composite micro-particles” to be a reference to intermetallic precipitate. Medlin Decl. Exhibit 2 at ¶ 256. As noted above, at least Fe, Cu, and Ni are “additive materials.” Consequently, the in-situ precipitation of galvanically- active intermetallic phases will include one or more of these additive materials.</p>
said magnesium composite has a dissolution rate of 84-325 mg/cm <sup>2</sup> /hr. in 3 wt % KCl water mixture at 90°C.	<p>A POSITA in April 2014 would have recognized that Xiao disclosed a table of dissolution rates (“decomposition rates”) because the units of measurement are the units used by POSITAs to measure dissolution rates. Medlin Decl. Exhibit 2 at ¶ 129. The disclosed table provides seven examples (excluding the comparative [prior art] example). Xiao at ¶ 0064. Xiao provides dissolution rates in g/cm<sup>2</sup>/hr, while the claims of the '653 Patent express dissolution rates in mg/cm<sup>2</sup>/hr. A POSITA would have known converting between g/cm<sup>2</sup>/hr to mg/cm<sup>2</sup>/hr, merely requires multiplication by 1,000. Medlin Decl. Exhibit 2 at ¶ 130.</p> <p>Thus, each of Xiao’s seven examples (excluding the comparative [prior art] example) has a dissolution rate within the claimed range of of at least three times the claimed “about 5-325 mg/cm<sup>2</sup>/hr in 3 wt.% KCl water” at both 70°C or 93°C (with Example 7 being more than seven times that floor), Dr. Medlin opines that a POSITA in April 2014 would have recognized that the dissolution rate of the examples in Xiao would necessarily have been well above the claimed floor of “about 5” “about 5-325 mg/cm<sup>2</sup>/hr” at the claimed 90°C. Id. at ¶ 132.</p> <p>Claim 1 of Xiao discloses a “magnesium alloy, comprising the components at the weight percentages as follows: Al: 13 to 25%, Zn: 2 to 15%, the remainder is Mg, and a sum of the</p>

	<p>weight percentages of the components is 100%.” Xiao (emphasis added). Dr. Medlin opines that a POSITA would have understood that the range of non-magnesium elements in Claim 1 leaves 60-85 wt% for magnesium. Medlin Decl. Exhibit 2 at ¶ 149. Thus, Xiao discloses a range of magnesium up to 85 wt%, which overlaps with the claimed range (i.e. at least 85wt% magnesium) set forth in this claim element.</p> <p>Xiao also discloses a prior art magnesium alloy represented by Mg-Al-Zn (with AZ91D magnesium alloy being the most widely used) wherein the “main components of this alloy and the respective weight percentages are as follows: Al 8.3 to 9.7, Zn 0.35 to 1.0, Mn 0.15 to 0.5, and the remainder [(i.e. 88.8 to 91.2 wt%)] is magnesium.” Id. at ¶ 0003; Medlin Decl. Exhibit 2 at ¶ 269. Xiao also uses an AZ91D as a comparative example alloy, with a composition of 9 wt% Al, 1 wt% Zn, 0.3 wt% Zr, and 0.1 wt% Mn, with the remainder (i.e. 89.6 wt%) being magnesium. Id. at ¶ 0032. Xiao teaches a primary difference between this prior art and its novel magnesium alloy lies in the addition of metallic elements like Iron, Copper, and/or Nickel into the alloy. Id. at ¶ 0026 (“[e]lements such as Fe, Cu, Ni, Ag, etc., in the magnesium alloy can form a large number of intermetallic composite micro-particles, which can improve the corrosion performance of the magnesium alloy, thereby promoting the decomposition of the magnesium alloy”). The dissolution data in Xiao (Xiao at ¶ 0061) teaches that the addition of Iron, Copper, and/or Nickel (alongside the Aluminum, Zinc, Zirconium, and Manganese of the prior art AZ91D example) caused a surprisingly large increase in dissolution rates.</p>
--	---

	<p>Thus, Xiao, as a whole, teaches magnesium compositions with magnesium content above 85wt% and “room” for the novel additive materials (i.e. “Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%” (Xiao at Claim 2)). Thus, Xiao fully discloses a range of magnesium of at least 85 wt %.</p> <p>As for the final limitation of Claim 74, it requires a dissolution rate of 84–325 mg/cm<sup>2</sup>/hr. in 3 wt % KCl water mixture at 90°C, as the discussion, supra, demonstrates, the novel examples explicitly analyzed in Xiao disclose dissolution rates only up to 74 mg/cm<sup>2</sup>/hr in 3 wt.% KCl water at 93°C, shy of this claimed range. Medlin Decl. Exhibit 2 at ¶ 330. However, Dr. Medlin opines that a POSITA in April 2014 would have understood Xiao to also teach dissolution rates above the floor of 84 mg/cm<sup>2</sup>/hr at 90°C claimed in Claim 74, because (1) the dissolution rates in the Xiao examples are very close to the claimed floor (e.g. Example 1 exhibited a rate of 74 mg/cm<sup>2</sup>/hr at 93°C, Example 6 a rate of 63 mg/cm<sup>2</sup>/hr at 90°C, and Example 7 a rate of 57 mg/cm<sup>2</sup>/hr at 93°C; and (2) a POSITA would have expected that other magnesium compositions having additive amounts in the ranges as disclosed by Xiao, but with higher amounts of copper, nickel, and/or iron than the amounts tested by Xiao’s examples would have improved corrosion performance. Medlin Decl. Exhibit 2 at ¶ 331. In particular, even though they come close to the 84 mg/cm<sup>2</sup>/hr at 90°C, Examples 1, 6 and 7 of Xiao had less aggregate copper, nickel and iron than taught by Claim 2 of Xiao (i.e. 5 wt% for each of copper, nickel, and iron in a magnesium composite). Medlin Decl. Exhibit 2 at ¶ 331. Thus, the Examples in Xiao used lower amounts of these specific dissolution-enhancing</p>
--	--

	<p>additive elements. Thus, according to Dr. Medlin, a POSITA in April 2014 would have understood this and recognized that Xiao teaches magnesium compositions having dissolution rates above the claimed floor of “at least 84 mg/cm<sup>2</sup>/hr.” Id. Dr. Medlin also notes that it was well-known prior to April 2014 that additions of copper, iron, nickel and cobalt cause a sharp linear increase in the corrosion rate of magnesium as evidenced by Shaw, “Corrosion Resistance of Magnesium Alloys” published in 2003 in the ASM Handbook, Vol. 13A (2003). Ex. 1204. Medlin Decl. Exhibit 2 at ¶ 332. As such, the recitation of the claimed dissolution rate of 84-325 mg/cm<sup>2</sup>/hr at 90°C was not a new property or use but rather a property that would be inherent in the compositions disclosed in Xiao, as confirmed by the extrinsic evidence of Shaw.</p>
<b>Dependent Claim 75</b>	<b>Disclosure of Claim Element in Xiao and/or Prior Art</b>
75. The magnesium composite as defined in claim 74, wherein said magnesium alloy is an AZ91D magnesium alloy that includes aluminum and zinc.	<p style="text-align: center;"><b>Magnesium Disclosed in Xiao</b></p> <p>Xiao discloses a “magnesium alloy...and further adds elements of Fe, Cu, Ni, and Ag which can enhance the corrosion performance of the magnesium alloy...” ¶ 0026, ll. 1-3.</p> <p>Claim 1 of Xiao discloses a “magnesium alloy, comprising the components at the weight percentages as follows: Al: 13 to 25%, Zn: 2 to 15%; the remainder is Mg, and a sum of the weight percentages of the components is 100%.” Claim 1. A POSITA would have understood that the total range of the non-magnesium elements disclosed in Claim 1 of Xiao is 15 to 40%, leaving 60-85 wt% for magnesium. Thus, Xiao discloses a range of magnesium 54.5-85 wt%, which overlaps and covers much of the claimed range.</p> <p>Additionally, ¶ 0003 discloses a known aluminum alloy:</p>

	The existing cast magnesium alloy is mainly represented by Mg-Al-Zn type magnesium alloys, and the AZ91D magnesium alloy is the most widely used. The main components of this alloy and the respective weight percentages are as follows: Al 8.3 to 9.7, Zn 0.35 to 1.0, Mn 0.15 to 0.5, and the remainder is magnesium. ¶ 0003.
<b>Dependent Claim 76</b>	<b>Disclosure of Claim Element in Xiao and/or Prior Art</b> <b>Magnesium Disclosed in Xiao</b>
76. The magnesium composite as defined in claim 74, wherein said additive material includes nickel,	Xiao teaches the use of a variety of metals as additive materials. See, e.g., ¶ 0026. For instance, Xiao claims that the novel magnesium alloy may “further compris[e] trace elements at the weight percentages as follows: Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%...” Id. at Claim 2. In addition, Example 7 of Xiao particularly discloses intermediate alloys Al-Fe, Al-Ni, Al-Ag, Al-Cu, Al-Zr, Al-Ti and Zinc as additive materials. Id. at ¶ 0060. A POSITA in April 2014 would have understood that all of these intermediate alloys contain metal, including, in particular, copper, nickel, and iron, among other metals. Thus, Xiao discloses a number of metal additives, including copper, nickel, and iron and, hence fully discloses all of the limitations of these claim elements.
a content of said nickel in said magnesium composite is at least 0.3 wt %.	Xiao teaches the use of a variety of metals as additive materials. See, e.g., ¶ 0026. For instance, Xiao claims that the novel magnesium alloy may “further compris[e] trace elements at the weight percentages as follows: Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%...” Id. at Claim 2. In addition, Example 7 of Xiao particularly discloses intermediate alloys Al-Fe, Al-Ni, Al-Ag, Al-Cu, Al-Zr, Al-Ti and Zinc as additive materials. Id. at ¶ 0060. A POSITA in April 2014 would have understood that all of these intermediate alloys contain metal, including, in particular, copper, nickel,

	and iron, among other metals. Thus, Xiao discloses a number of metal additives, including copper, nickel, and iron and, hence fully discloses all of the limitations of these claim elements.
--	---

iii. **Ground III: Claims 7, 12–16, 18–21, 23–24, and 71 are Obvious over Xiao in combination with Hassan**

i. **Obviousness**

A party that requests a determination of obviousness must show that “a skilled artisan would have been motivated to combine the teachings of the prior art references to achieve the claimed invention, and that the skilled artisan would have had a reasonable expectation of success in doing so.” *Apotex Inc. v. Wyeth LLC*, IPR2014-00115, Paper 94, slip op. at 11 (Apr. 20, 2015) (quoting *Procter & Gamble Co. v. Teva Pharm. USA, Inc.*, 566 F.3d 989, 994 (Fed. Cir. 2009)). The obviousness determination turns on underlying factual inquiries involving: (1) the scope and content of prior art, (2) differences between claims and prior art, (3) the level of ordinary skill in pertinent art, and (4) secondary considerations such as commercial success and satisfaction of a long-felt need. *Procter & Gamble Co. v. Teva Pharm. USA, Inc.*, 566 F.3d 989, 994 (Fed. Cir. 2009) (quoting *Graham v. John Deere Co.*, 383 U.S. 1, 17, 86 S.Ct. 684, 15 L.Ed.2d 545 (1966)).

ii. **Motivation to combine Xiao and Hassan**

Dr Medlin opines that a POSITA in April 2014 would have been motivated to combine the disclosures of Xiao with the teachings of Hassan. Ex. 1002 at ¶¶ 337- 344. First, Xiao and Hassan are both directed to improving the properties of magnesium based materials, especially those for use in devices or tools. *Id.* Hassan, for instance, teaches that:

increasing hostile service conditions that the modern engineering devices have to withstand have led the materials scientists across the globe to create new materials with enhanced properties when compared to the conventional materials. One way to improve the properties of conventional metallic

materials is to reinforce them judiciously keeping the end application in mind. Among the reinforced metallic materials, magnesium based composites are becoming the strong candidates for lightweight structural application due to their superior specific mechanical properties.

Ex. 1007 at 2467. Hassan also recognizes that type of processing, type, size, and volume fraction of reinforcement are “common factors governing the end properties of a metal matrix composite[].” *Id.*

Xiao similarly teaches in the context of its smelt casted magnesium alloy, the use of reinforcing metals or metal alloys to enhance corrosion performance and/or improve the compressive strength of the formed magnesium material. Ex. 1006 at ¶ 0026. In particular, Xiao noted that existing magnesium alloys (such as AZ91D) had low compressive strength and slow decomposition rates, making them unsuitable as material for, for example, a tripping ball used in oil well drilling. *Id.* at ¶ 0003. Xiao responded to those recognized problems in the art by “adjusting the chemical composition and preparation process” to prepare a light and pressure-proof cast magnesium alloy that adds elements which can enhance the corrosion performance of the magnesium alloy. *Id.* at ¶ 0004.

Dr. Medlin further opined that a POSITA would have been motivated to modify the disclosed magnesium alloys and magnesium-alloy-based tripping ball material taught in Xiao to further increase and/or control the rate of dissolution. Ex. 1002 at ¶ 340. More specifically, Dr. Medlin opined that a POSITA would look to Hassan, which teaches pure magnesium reinforced with nickel particulates that exhibits improved physical and mechanical properties over magnesium alloy AZ91. *Id.* at ¶ 341 (citing Ex. 1007 at p. 2467, col. 2). Hassan also teaches that careful “selection of reinforcing phase remains one of the most critical factors in developing a composite material with superior properties when compared to its monolithic counterparts.” *Id.* Additionally, Medlin said that Hassan notes that its magnesium composites possess improved

thermal expansion and mechanical characteristics, which would be beneficial in oil field applications. *Id.* Accordingly, Dr. Medlin concludes that a POSITA in April 2014 would have recognized the benefits set forth in Hassan applied to the magnesium alloy used in the tools taught in Xiao. *Id.*

Hassan also teaches subjecting magnesium ingots to secondary processing. Dr. Medlin opines that a POSITA in April 2014 would have been motivated to treat the magnesium alloys disclosed in Xiao using the secondary processing techniques disclosed by Hassan such that the size of additive particulates and magnesium composite could be controlled and optimized. Ex. 1002 at ¶ 344.

Xiao already disclosed that controlling the particulate size of its magnesium alloy was desirable with argon treatment and a refining agent (rather than the deformation processing claimed in the ‘653 Patent). Ex. 1006 at ¶¶ 0036 and 0044. Dr. Medlin opined that a POSITA in April 2014 would have had a reasonable expectation of successfully combining the magnesium alloy of Xiao with the secondary processing techniques of Hassan, which were conventionally techniques known for decades to optimize particulate size of metal alloys and improve metal properties. Ex. 1002 at ¶ 343. Moreover, such processing techniques have been known for decades to optimize particulate size of metal alloys. See, e.g., Ex. 1203, 1:20-23 (“Powder metal technology is well known to the persons skilled in the art and generally comprises the formation of metal powders which are compacted and then subjected to an elevated temperature so as to produce a sintered product.”).

**iii. Independent Claim 12 and Dependent Claims 7, 13–16, 18–21, 23, 24, and 71 are Obvious over Xiao in combination with Hassan**

A POSITA reading the disclosure of Xiao in combination with the disclosures of Hassan would have found obvious Claims 7, 12–16, 18–21, 23, 24, and 71 of the ‘653 Patent based on the following disclosures and motivation to combine Xiao and Hassan:

<b><u>'653 Patent</u></b> <b><u>Claims/Elements</u></b>	<b><u>Made Obvious by Disclosures of Xiao in Combination with Hassan</u></b>
<b>Dependent Claim 7</b>	<b>Disclosure of Claim Element in Xiao and/or in Combination with Hassan</b>
7. The magnesium composite as defined in claim 1, where said magnesium composite is subjected to a deformation processing to reduce grain size of said magnesium composite, increase tensile yield strength of said magnesium composite, increase elongation of said magnesium composite, or combinations thereof.	<p>As set forth in Ground I, <i>supra</i>, Xiao already discloses each and every element of the magnesium composition of Claim 1 from which Claim 7 depends. Xiao does not disclose subjecting that magnesium composite to deformation processing.</p> <p>Hassan explicitly discloses deformation processing. In particular, Hassan discloses that the deposited magnesium ingots may then be subjected to secondary processing. For example, Hassan teaches that the nickel-reinforced magnesium ingots are machined and hot extruded to obtain rods of 8 mm diameter. <i>Id.</i> Analysis of the composite of Hassan “showed the reduction in the size of elemental nickel particulates in the extruded composites” (see Table 1 (from Hassan (above))). In other words, the extrusion in Hassan resulted in a reduction of grain size of the magnesium composite.</p> <p>A POSITA in April 2014 would have been motivated to combine the disclosure of Xiao with this disclosure of deformation processing in Hassan rendering Claim 7 obvious. Accordingly, Claim 7 should be found obvious over Xiao in view of Hassan.</p>

Independent Claim 12	Disclosure of Claim Element in Xiao and/or in Combination with Hassan
<p>12. A magnesium composite that includes in situ precipitation of galvanically-active intermetallic phases to enable controlled dissolution of said magnesium composite comprising a mixture of a magnesium or a magnesium alloy and an additive material,</p>	<p>Xiao discloses a “magnesium alloy...and further adds elements of Fe, Cu, Ni, and Ag which can enhance the corrosion performance of the magnesium alloy...” ¶ 0026, ll. 1-3.</p> <p>Xiao discloses a novel magnesium alloy with high aluminum and zinc content further adding elements Fe, Cu, Ni and Ag which can form intermetallics and enhance the corrosion performance of the magnesium alloy. Xiao explains the corrosion mechanism within a magnesium alloy in terms of a cathode phase, which Xiao explains includes “the <math>\beta</math> (<math>Mg_{17}Al_{12}</math>) phase and eutectic phase on the grain boundary of the magnesium alloy,” and “the magnesium matrix <math>\alpha</math> phase which functions as an anode phase, so that the matrix and the grain boundary of the magnesium alloy form a large amount of micro-batteries, which [micro-batteries] greatly accelerate the corrosion decomposition of magnesium alloy.” ¶ 0026.</p> <p>A POSITA in August 2014 would have understood this disclosure of “intermetallic composite micro-particles” to be a reference to intermetallic precipitate.</p> <p>A POSITA reading the Xiao reference in April 2014 would have recognized that the precipitation of galvanically-active intermetallic phases (“micro-batteries”) are naturally and necessarily generated by the in situ reaction between magnesium and any and all of the disclosed metal additives forming in situ precipitate. Thus, this claim element was disclosed by Xiao.</p> <p>Xiao discloses “first loading pure magnesium and pure aluminum into a smelting furnace . . . next loading pure zinc and an [sic] intermediate alloys of trace element components into a</p>

	<p>resulting magnesium-aluminum alloy melt after melting.” ¶ 0022. Xiao discloses that “Al-Fe ... Al-Ni ... Al-Cu ... Al-Ag ... Al-Zr ... and ... Al-Ti intermediate alloy[s], are heated to dry and then added to the magnesium-aluminum alloy melt.” Id. at ¶ 0023. The addition of these intermediate alloys to the melted magnesium alloy necessarily results in a mixture of the additive material and magnesium alloy.</p>
said additive material having a greater melting point temperature than a solidus temperature of said magnesium,	<p>Using a typical phase diagram, a POSITA in April 2014 would have readily determined that the solidus temperature of magnesium is the temperature at which a metal first begins to melt during heating and not necessarily when the metal is completely liquidus.</p> <p>RESPECTIVE DIAGRAM OMITTED</p> <p>Ex. 1213. A POSITA would also use this phase diagram to find the solidus temperature of the magnesium-aluminum alloy disclosed in Xiao to be in a range of 650°C for pure magnesium and down to 437°C for aluminum additions in the range of 13 wt% to 25% and even up to 40 wt%.</p> <p>The POSITA using typical phase diagrams for each of the additive materials (here the Al-Fe, Al-Ni, Al-Cu, Al-Ag, Al-Zr, Al-Ti “intermediate alloys” disclosed in Xiao) would be able to readily determine the melting point temperature for each intermediate alloy. (¶ 0023). For example, the Al-Fe phase diagram (Ex. 1205) shows a variable melting point temperature ranging from 1538°C (pure iron) down to 655°C for a eutectic temperature between about 63 wt% aluminum to near 99 wt% aluminum. The Al-Ni phase diagram (Ex. 1206) shows a variable melting point temperature that ranges from 1640°C down to 640°C depending upon the alloy content. The Al-Cu (Ex. 1207) phase diagram shows a variable melting point temperature ranging from 548°C up to 1084°C depending upon</p>

the alloy mixture. A POSITA in April 2014 would have readily determined the specific melting temperature of a specific alloy mixture based on these phase diagrams.

Xiao discloses a variety of Al-Fe, Al-Ni, Al-Cu, Al-Ag, Al-Zr, Al-Ti alloy mixtures with a variety of constituencies. For instance, Xiao specifically teaches in Example 7 that:

The composition of the alloy and the respective percentages by weight are as follows: 25% Al-10% Zn-1% Fe-0.5% Ni-0.1% Cu- 0.5% Ti-0.05% Zr, and the remainder is Mg.

¶ 0058-59. Xiao teaches that the magnesium alloy of Example 7, as a whole, ends up with the maximum amount of aluminum (i.e. 25 wt%) taught as being within the invention of Xiao (see, ¶ 0026). Xiao specifically discloses—with respect to Example 7—that portions of that aluminum content are added into a magnesium-aluminum melt as an Al-Fe intermediate alloy, an Al-Ni intermediate alloy, an Al-Cu intermediate alloy, an Al-Ag intermediate alloy, an Al-Zr intermediate alloy, and an Al-Ti intermediate alloy. ¶ 0060. By converting weight percentages to grams and calculating mass balances, a POSITA would first determine the relative concentrations of the pure magnesium and pure aluminum initially loaded into the smelting furnace in order to determine its solidus temperature. As a result, a POSITA would have understood in April 2014 that Example 7 of Xiao includes 62.85 total grams of magnesium.

The following intermediate alloy compositions were chosen for the initial mass balance calculations: Al-Fe (30 wt% Fe), Al-Ni (45 wt% Ni), Al-Cu (40 wt% Cu), Al-Ti (10 wt% Ti), Al-Zr (20 wt% Zr) alloy mixtures. A POSITA in April 2014 would have performed the initial mass balances, included herewith as the Mass/Balance Exhibit 2, on these compositions. Using these initially selected alloy compositions would have resulted in 7.79

grams of Aluminum being added as components of the intermediate alloys. Since Example 7 of Xiao calls for a total of 25 grams (i.e. 25 wt%) of Aluminum, 17.21 grams of Aluminum would have had to have been initially loaded into the smelting furnace in Example 7 along with the 62.85 grams of magnesium (see calculation above). Thus, the initial melt of Example 7 (using the intermediate alloys selected) would have had a composition of 78.5 wt% Mg and 21.5 wt% Al.

A POSITA in April 2014 would have next consulted the Mg-Al phase diagram to determine that the solidus temperature of that initial magnesium alloy (i.e. 78.5 wt% Mg and 21.5 wt% Al) would be 437 °C. A POSITA would also consult the relevant phase diagram to determine the melting point temperature of the various additive materials disclosed in Xiao. Id. at ¶ 122. An aluminum-iron alloy with 30 wt% iron would have a melting point temperature of 655 °C, an aluminum-nickel alloy with 45 wt% Ni would have a melting point temperature of 854 °C, an aluminum-copper alloy with 40 wt% Cu would have a melting point temperature of 548°C, an aluminum-titanium alloy with 10 wt% Ti would have a melting point temperature of 665°C, and an aluminum-zirconium alloy with 20 wt% Zr would have a melting point temperature of 661°C. Thus, each of these additive materials from Example 7 of Xiao (with the constituencies selected above) would have had a greater melting point temperature than the solidus temperature (i.e. 437°C) of the magnesium-aluminum alloy (78.5 wt% Mg and 21.5 wt% Al) calculated as the amount of pure aluminum and magnesium initially loaded into the smelting furnace in Example 7.

A POSITA in April 2014 would have recognized that other compositions would have fallen within the teachings of Example 7 and other disclosures of Xiao resulting in Al-Fe, Al-

	Ni, and Al-Cu intermediate alloys having greater melting point temperatures than the solidus temperature of initial magnesium alloy loaded in the various disclosures of Xiao. Thus, Xiao discloses all of the limitations of this claim element.
said composite including greater than 50 wt % magnesium,	Claim 1 of Xiao discloses a “magnesium alloy, comprising the components at the weight percentages as follows: Al: 13 to 25%, Zn: 2 to 15%; the remainder is Mg, and a sum of the weight percentages of the components is 100%.” Claim 1. Thus, Xiao discloses a range of magnesium 54.5-85 wt%, which overlaps and covers much of the claimed ranges set forth in these claim elements.
said additive material constituting about 0.05-45 wt % of said magnesium composite,	Claim 1 of Xiao discloses: a “magnesium alloy, comprising the components at the weight percentages as follows: Al: 13 to 25%, Zn: 2 to 15%, the remainder is Mg, and a sum of the weight percentages of the components is 100%.” Claim 2 discloses: a “magnesium alloy, according to Claim 1, further comprising the trace elements at the weight percentages as follows: Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%; and a sum of the weight percentages of the components is 100%.” These metallic elements are found in the additive materials generally disclosed by Xiao (i.e., intermediate alloys: Al-Fe, Al-Ni, Al-Cu, Al-Ti) (¶ 0023) and particularly described in association with Example 7 of Xiao (Id. at ¶¶ 0057-0060) for which a POSITA could calculate the specific weight percentage (ranges) of each additive material or secondary metal. In doing so, a POSITA would determine that the additive materials aluminum-iron with 30 wt% iron, aluminum-nickel with 45 wt% nickel, and aluminum-copper with 40 wt% copper also fall well within the range of about 0.05 wt% to 45 wt% of the mixture. For example, the aluminum-iron additive constitutes

	<p>3.3 wt% of the mixture; the aluminum-nickel additive constitutes 1.1 wt% of the mixture; and the aluminum-copper additive constitutes 0.25 wt% of the mixture. A POSITA in April 2014 would have also recognized that other intermediate alloy compositions would have fallen within the teachings of Example 7 and other disclosures of Xiao resulting in Al-Fe, Al-Ni, and Al-Cu intermediate alloys constituting about 0.05 wt% to 45 wt% of the mixture. Accordingly, this claim element was disclosed by Xiao.</p>
said additive material having a melting point temperature that is 100°C. greater than a melting temperature of said magnesium or magnesium alloy,	<p>A POSITA in April 2014 would have understood “melting temperature” in this claim element to be a reference to the smelting temperature or temperature of the melt when the additive material is being added to the magnesium or magnesium alloy, not the “melting point temperature” as that measurement is used for the additive material.</p> <p>Xiao generally discloses “loading pure magnesium and pure aluminum into a smelting furnace and increasing the temperature to 700 to 730 °C, next loading pure zinc and an [sic] intermediate alloys . . . into a resulting magnesium-aluminum alloy melt after melting and increasing the temperature to 740 to 780°C . . .” ¶¶ 0021–0022; see also Id. at Claim 5 (“the melting temperature of the pure magnesium and pure aluminum is from 700 to 730 °C.”)</p> <p>Examples 1–7 of Xiao teach a range of smelting temperatures for the magnesium-aluminum alloy from 700°C (Example 1) to 730°C (Examples 2, 5, and 7). ¶¶ 0034–0060.</p> <p>The additive materials noted above are one or more of the intermediate alloys: Al-Fe, Al-Ni, Al-Cu, Al-Ag, Al-Zr, Al-Ti. Id. at ¶ 0023. A POSITA using typical phase diagrams for each of these additive materials would be able to readily determine the melting point temperature.</p>

	<p>Example 7 of Xiao specifically discloses a melting or smelting temperature of 730°C. ¶ 0060. A POSITA in April 2014 would have recognized from Example 7 of Xiao that at least an aluminum-nickel additive having 45 wt% nickel has a melting point temperature of 854°C which is 100°C greater than the melting or smelting temperatures of the magnesium aluminum alloy used in Example 7 (i.e., 730°C) and otherwise taught generally by Xiao (i.e., 700-730°C). A POSITA in April 2014 would have also recognized that other intermediate alloy compositions could have fallen within the teachings of Example 7 and other disclosures of Xiao and resulted in Al-Fe, Al-Ni, and Al-Cu intermediate alloys that would have had melting point temperatures that were 100°C. greater than the smelting temperatures of the magnesium alloy generally taught by Xiao (i.e., 700- 730°C). Thus, this claim element was disclosed by Xiao.</p>
said additive material including one or more metals selected from the group consisting of copper, nickel, cobalt, titanium, and iron,	<p>Xiao teaches the use of a variety of metals as additive materials. See, e.g., ¶ 0026. For instance, Xiao claims that the novel magnesium alloy may “further compris[e] trace elements at the weight percentages as follows: Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%. . .” Id. at Claim 2. In addition, Example 7 of Xiao particularly discloses intermediate alloys Al-Fe, Al-Ni, Al-Ag, Al-Cu, Al-Zr, Al-Ti and Zinc as additive materials. Id. at ¶ 0060. A POSITA in April 2014 would have understood that all of these intermediate alloys contain metal, including, in particular, copper, nickel, and iron, among other metals. Thus, Xiao discloses a number of metal additives, including copper, nickel, and iron and, hence fully discloses all of the limitations of these claim elements.</p>

<p>at least a portion of said additive material remaining unalloyed additive material,</p>	<p>Xiao discloses that “[e]lements such as Fe, Cu, Ni, Ag, etc., in the micro-particles.” ¶ 0026. However, Xiao does not disclose whether any of the additive material remains “unalloyed”. Hassan explicitly discloses that magnesium composites containing additive materials, such as nickel may remain unreacted. In particular, Hassan discloses that the [s]evere reaction between magnesium melt and nickel particulates during DMD processing led to the reduction of the particulate size and formation of Mg<sub>2</sub>Ni intermetallics [13]. The results of quantitative determination of unreacted nickel (see Table I), microstructural characterization illustrating the presence of reaction products (see Fig. 1) and XRD results (see Table II) showing the presence of Mg<sub>2</sub>Ni supports the experimental observations.” Thus, Hassan specifically provides experimental data showing— under similar conditions of magnesium composite fabrication as those in Xiao—that some of the additives, particularly nickel, will remain unreacted and unalloyed.</p> <p>Given the motivation to combine Xiao with Hassan, a POSITA in April 2014 would have combined these two references thereby providing further rationale that a POSITA in April 2014 would find Claim 12 of the ‘653 Patent obvious.</p>
<p>said magnesium composite including in situ precipitation of galvanically-active intermetallic phases that includes said unalloyed additive material,</p>	<p>Xiao discloses a novel magnesium alloy with high aluminum and zinc content further adding elements Fe, Cu, Ni and Ag which can form intermetallics and enhance the corrosion performance of the magnesium alloy. ¶ 0026. Xiao explains the corrosion mechanism within a magnesium alloy in terms of a cathode phase, which Xiao explains includes “the <math>\beta</math> (Mg<sub>17</sub>Al<sub>12</sub>) phase and eutectic phase on the grain boundary of the magnesium alloy,” and “the magnesium matrix <math>\alpha</math> phase</p>

which functions as an anode phase, so that the matrix and the grain boundary of the magnesium alloy form a large amount of micro-batteries, which [micro-batteries] greatly accelerate the corrosion decomposition of magnesium alloy.” Id.

A POSITA in August 2014 would have understood this disclosure of “intermetallic composite micro-particles” to be a reference to intermetallic precipitate. As noted above, at least Fe, Cu, and Ni are “additive materials.” Consequently, the in-situ precipitation of galvanically-active intermetallic phases will include one or more of these additive materials. Thus, Xiao discloses all of this claim limitation.

Xiao discloses that “[e]lements such as Fe, Cu, Ni, Ag, etc., in the micro- particles.” Id. at ¶ 0026. However, Xiao does not disclose whether any of the additive material remains “unalloyed”.

Hassan explicitly discloses that magnesium composites containing additive materials, such as nickel may remain unreacted. In particular, Hassan discloses that the [s]evere reaction between magnesium melt and nickel particulates during DMD processing led to the reduction of the particulate size and formation of Mg<sub>2</sub>Ni intermetallics [13]. The results of quantitative determination of unreacted nickel (see Table I), microstructural characterization illustrating the presence of reaction products (see Fig. 1) and XRD results (see Table II) showing the presence of Mg<sub>2</sub>Ni supports the experimental observations.”

Ex. 1007 at 2472. Thus, Hassan specifically provides experimental data showing— under similar conditions of magnesium composite fabrication as those in Xiao—that some

	<p>of the additives, particularly nickel, will remain unreacted and unalloyed.</p> <p>Given the motivation to combine Xiao with Hassan (discussed, <i>supra</i>), Dr. Medlin opined that a POSITA in April 2014 would have combined these two references thereby providing further rationale that a POSITA in April 2014 would find Claims 12 and 71 of the '653 Patent obvious.</p>
said magnesium composite has a dissolution rate of at least 5 mg/cm <sup>2</sup> /hr. in 3 wt % KCl water mixture at 90°C.	<p>A POSITA in April 2014 would have recognized that Xiao disclosed a table of dissolution rates ("decomposition rates") because the units of measurement are the units used by POSITAs to measure dissolution rates. The disclosed table provides seven examples (excluding the comparative [prior art] example). ¶ 0064.</p> <p>Xiao provides dissolution rates in g/cm<sup>2</sup>/hr, while the claims of the '653 Patent express dissolution rates in mg/cm<sup>2</sup>/hr. A POSITA would have known converting between g/cm<sup>2</sup>/hr to mg/cm<sup>2</sup>/hr, merely requires multiplication by 1,000.</p> <p>Thus, each of Xiao's seven examples (excluding the comparative [prior art] example) has a dissolution rate of at least three times the claimed "at least 5 mg/cm<sup>2</sup>/hr in 3 wt.% KCl water" at both 70°C or 93°C (with Example 7 being more than seven times that floor). A POSITA in April 2014 would have recognized that the dissolution rate of the examples in Xiao would necessarily have been well above the claimed floor of "at least of 5 mg/cm<sup>2</sup>/hr" at the claimed 90°C.</p>
Dependent Claim 13	<b>Disclosure of Claim Element in Xiao and/or in Combination with Hassan</b>
13. The magnesium composite as defined in claim 12, wherein said additive	With respect to the additional limitations added by this dependent claim, Xiao discloses "first loading pure magnesium and pure aluminum into a smelting furnace, next loading pure

<p>material is added to said magnesium or magnesium alloy while said magnesium or magnesium alloy is at a temperature that is above said solidus temperature of said magnesium and a temperature that is less than a melting point of said additive material to form said mixture.</p>	<p>zinc and an [sic] intermediate alloys . . . into a resulting magnesium-aluminum alloy melt after melting.” Xiao at Claim 4.</p> <p>Hassan, discloses, in particular, magnesium as a base material “and elemental nickel particulates of 99.9% purity (Johnson Matthey, MA, USA) with an average size of <math>29\pm19 \mu\text{m}</math> were used as reinforcement phase” and that “[t]he synthesis of the composites involved superheating the magnesium turnings with reinforcement particulates (placed in multi-layer sandwich form) to <math>750^\circ\text{C}</math> under inert Ar gas atmosphere in a graphite crucible.” Ex. 1007 at p. 2468.</p> <p>A POSITA would have known in April 2014 that the melting point of pure magnesium is <math>650^\circ\text{C}</math> and the melting point of pure nickel is <math>1455^\circ\text{C}</math> using standard phase diagrams for Mg and Ni, respectively. Medlin Decl. Exhibit 2 at ¶ 51. Given these values and the disclosure in Hassan of heating only to <math>750^\circ\text{C}</math>, the combination of Xiao and Hassan necessarily teach the limitations of Claim 13.</p>
Dependent Claim 14	Disclosure of Claim Element in Xiao and/or in Combination with Hassan
<p>14. The magnesium composite as defined in claim 13, wherein said magnesium alloy includes over 50 wt % magnesium and one or more metals selected from the group consisting of aluminum in an amount of about 0.5-10 wt %, zinc in amount of about 0.1-3 wt %, zirconium in an amount of</p>	<p>Xiao discloses a “magnesium alloy...and further adds elements of Fe, Cu, Ni, and Ag which can enhance the corrosion performance of the magnesium alloy...” Xiao at ¶ 0026, ll. 1-9.</p> <p>Claim 1 of Xiao discloses a “magnesium alloy, comprising the components at the weight percentages as follows: Al: 13 to 25%, Zn: 2 to 15%; the remainder is Mg, and a sum of the weight percentages of the components is 100%.” Xiao at Claim 1 (emphasis added). Dr. Medlin opines that a POSITA would have understood that the total range of the non-magnesium elements disclosed in Claim 1 of Xiao is 15 to 40%, leaving 60-</p>

<p>about 0.01-1 wt %, manganese in an amount of about 0.15-2 wt %, boron in amount of about 0.0002-0.04 wt %, and bismuth in an amount of about 0.4-0.7 wt %.</p>	<p>85 wt% for magnesium. Medlin Decl. Exhibit 2 at ¶ 149. Dr. Medlin also notes that the “Examples of Xiao disclose a range of magnesium from 54.5 wt% - 79.2 wt%.” Id. Thus, Xiao discloses a range of magnesium 54.5-85 wt%, which overlaps and covers much of the claimed range. This claim element would be anticipated solely by the zirconium concentrations falling within the 0.01-1 and/or 0.01-3 wt% ranges. Claim 14 also requires, in addition to a similar zirconium concentration to the Markush claims, a zinc concentration of 0.1-3 wt%.</p>
<b>Dependent Claim 15</b>	<b>Disclosure of Claim Element in Xiao and/or in Combination with Hassan</b>
<p>15. The magnesium composite as defined in claim 14, wherein said additive material includes nickel,</p>	<p>Xiao discloses a “magnesium alloy...and further adds elements of Fe, Cu, Ni, and Ag which can enhance the corrosion performance of the magnesium alloy...” Xiao at ¶ 0026, ll. 1-9. Xiao teaches the use of a variety of metals as additive materials. See, e.g., Xiao at ¶ 0026. For instance, Xiao claims that the novel magnesium alloy may “further compris[e] trace elements at the weight percentages as follows: Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%...”Id. at Claim 2. In addition, Example 7 of Xiao particularly discloses intermediate alloys Al-Fe, Al-Ni, Al-Ag, Al-Cu, Al-Zr, Al-Ti and Zinc as additive materials. Id. at ¶ 0060. A POSITA in April 2014 would have understood that all of these intermediate alloys contain metal, including nickel, among other metals. Medlin Decl. Exhibit 2 at ¶ 145. Thus, Xiao discloses a number of metal additives, including nickel, and hence fully discloses all of the limitations of these claim elements.</p>

<p>said nickel constitutes about 0.05-35 wt % of said magnesium composite,</p>	<p>Claim 1 of Xiao discloses: a “magnesium alloy, comprising the components at the weight percentages as follows: Al: 13 to 25%, Zn: 2 to 15%, the remainder is Mg, and a sum of the weight percentages of the components is 100%.” Xiao at Claim 1. And Claim 2 discloses: a “magnesium alloy, according to Claim 1, further comprising the trace elements at the weight percentages as follows: Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%; and a sum of the weight percentages of the components is 100%.” Id. at Claim 2.</p> <p>These metallic elements are found in the additive materials generally disclosed by Xiao (i.e., intermediate alloys: Al-Fe, Al-Ni, Al-Cu, Al-Ti) (Xiao at ¶ 0023) and particularly described in association with Example 7 of Xiao (Id. at ¶¶ 0057–0060) for which Dr. Medlin calculated for Example 7 of Xiao (Medlin Decl. Exhibit 2 at ¶¶ 113–122) the specific weight percentage (ranges) of each additive material or secondary metal. In particular, Dr. Medlin determined that the additive materials aluminum-iron with 30 wt% iron, aluminum-nickel with 45 wt% nickel, and aluminum-copper with 40 wt% copper also fall well within the range of about 0.05 wt% to 45 wt% of the mixture. Id. ¶ 122. For example, the aluminum-iron additive constitutes 3.3 wt% of the mixture; the aluminum-nickel additive constitutes 1.1 wt% of the mixture; and the aluminum-copper additive constitutes 0.25 wt% of the mixture. Dr. Medlin expressed his opinion that a POSITA in April 2014 would have also recognized that other intermediate alloy compositions would have fallen within the teachings of Example 7 and other disclosures of Xiao resulting in Al-Fe, Al-Ni, and Al-Cu intermediate alloys constituting about 0.05 wt% to 45 wt% of</p>
--	---

	the mixture. Id. at ¶ 125. Accordingly, this claim element was disclosed by Xiao.
said nickel forms galvanically-active in situ precipitate in said magnesium composite.	Xiao discloses a novel magnesium alloy with high aluminum and zinc content further adding elements Fe, Cu, Ni and Ag which can form intermetallics and enhance the corrosion performance of the magnesium alloy. Xiao at ¶ 0026. Xiao explains the corrosion mechanism within a magnesium alloy in terms of a cathode phase, which Xiao explains includes “the $\beta$ ( $Mg_{17}Al_{12}$ ) phase and eutectic phase on the grain boundary of the magnesium alloy,” and “the magnesium matrix $\alpha$ phase which functions as an anode phase, so that the matrix and the grain boundary of the magnesium alloy form a large amount of micro-batteries, which [micro-batteries] greatly accelerate the corrosion decomposition of magnesium alloy.” Id. These elements are among the claimed “secondary metals” disclosed by Xiao. Thus, all of the limitations of this claim element are disclosed by Xiao.
<b>Dependent Claim 16</b>	<b>Disclosure of Claim Element in Xiao and/or in Combination with Hassan</b>
16. The magnesium composite as defined in claim 15, wherein said additive material includes nickel,	Xiao discloses a “magnesium alloy...and further adds elements of Fe, Cu, Ni, and Ag which can enhance the corrosion performance of the magnesium alloy...” Xiao at ¶ 0026, ll. 1–9. Xiao teaches the use of a variety of metals as additive materials. See, e.g., Xiao at ¶ 0026. For instance, Xiao claims that the novel magnesium alloy may “further compris[e] trace elements at the weight percentages as follows: Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%...” Id. at Claim 2. In addition, Example 7 of Xiao particularly discloses intermediate alloys Al-Fe, Al-Ni, Al-Ag, Al-Cu, Al-Zr, Al-Ti and Zinc as additive materials. Id. at ¶ 0060. A POSITA in April

	2014 would have understood that all of these intermediate alloys contain metal, including nickel, among other metals. Medlin Decl. Exhibit 2 at ¶ 145. Thus, Xiao discloses a number of metal additives, including nickel, and hence fully discloses all of the limitations of these claim elements.
said nickel constitutes about 3-7 wt % of said magnesium composite.	<p>Claim 1 of Xiao discloses: a “magnesium alloy, comprising the components at the weight percentages as follows: Al: 13 to 25%, Zn: 2 to 15%, the remainder is Mg, and a sum of the weight percentages of the components is 100%.” Xiao at Claim 1. And Claim 2 discloses: a “magnesium alloy, according to Claim 1, further comprising the trace elements at the weight percentages as follows: Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%; and a sum of the weight percentages of the components is 100%.” Id. at Claim 2.</p> <p>These metallic elements are found in the additive materials generally disclosed by Xiao (i.e., intermediate alloys: Al-Fe, Al-Ni, Al-Cu, Al-Ti) (Xiao at ¶ 0023) and particularly described in association with Example 7 of Xiao (Id. at ¶¶ 0057–0060) for which Dr. Medlin calculated for Example 7 of Xiao (Medlin Decl. Exhibit 2 at ¶¶ 113–122) the specific weight percentage (ranges) of each additive material or secondary.</p> <p>In particular, Dr. Medlin determined that the additive materials aluminum-iron with 30 wt% iron, aluminum-nickel with 45 wt% nickel, and aluminum-copper with 40 wt% copper also fall well within the range of about 0.05 wt% to 45 wt% of the mixture. Id. ¶ 122. For example, the aluminum-iron additive constitutes 3.3 wt% of the mixture; the aluminum-nickel additive constitutes 1.1 wt% of the mixture; and the aluminum-copper additive constitutes 0.25 wt% of the mixture. Dr. Medlin</p>

	expressed his opinion that a POSITA in April 2014 would have also recognized that other intermediate alloy compositions would have fallen within the teachings of Example 7 and other disclosures of Xiao resulting in Al-Fe, Al-Ni, and Al-Cu intermediate alloys constituting about 0.05 wt% to 45 wt% of the mixture. Id. at ¶ 125. Accordingly, this claim element was disclosed by Xiao.
<b>Dependent Claim 18</b>	<b>Disclosure of Claim Element in Xiao and/or in Combination with Hassan</b>
18. The magnesium composite as defined in claim 15, wherein a dissolution rate of said magnesium composite is at least 45 mg/cm <sup>2</sup> /hr. in 3 wt % KCl water mixture at 90°C. and up to 325 mg/cm <sup>2</sup> /hr. in 3 wt % KCl water mixture at 90°C.	<p>A POSITA in April 2014 would have recognized that Xiao disclosed a table of dissolution rates (“decomposition rates”) because the units of measurement are the units used by POSITAs to measure dissolution rates. Medlin Decl. Exhibit 2 at ¶ 129. The disclosed table provides seven examples (excluding the comparative [prior art] example). Xiao at ¶ 0064.</p> <p>Xiao provides dissolution rates in g/cm<sup>2</sup>/hr, while the claims of the '653 Patent express dissolution rates in mg/cm<sup>2</sup>/hr. A POSITA would have known converting between g/cm<sup>2</sup>/hr to mg/cm<sup>2</sup>/hr, merely requires multiplication by 1,000. Medlin Decl. Exhibit 2 at ¶ 130.</p> <p>Six of the seven examples (excluding the comparative [prior art] example) provided in Xiao therefore discloses a dissolution rate of “at least 45 mg/cm<sup>2</sup>/hr in 3 wt % KCl water” and “up to 325 mg/cm<sup>2</sup>/hr in 3 wt % KCl water” at 93°C. Given that the dissolution rates of Xiao slightly above (i.e., 3°C above) 90°C are almost all above the claimed floor of “at least 45 mg/cm<sup>2</sup>/hr”. As such and per Dr. Medlin’s opinion, a POSITA in April 2014 would have expected the dissolution rate of the examples in Xiao at 90°C to also be necessarily above the</p>

	claimed floor of “at least 45 mg/cm <sup>2</sup> /hr.” Medlin Decl. Exhibit 2 at ¶ 205. Thus, this element is disclosed by Xiao.
<b>Dependent Claim 19</b>	<b>Disclosure of Claim Element in Xiao and/or in Combination with Hassan</b>
19. The magnesium composite as defined in claim 12, wherein said magnesium alloy includes over 50 wt % magnesium and one or more metals selected from the group consisting of aluminum in an amount of about 0.5-10 wt %, zinc in amount of about 0.1-6 wt %, zirconium in an amount of about 0.01-3 wt %, manganese in an amount of about 0.15-2 wt %, boron in amount of about 0.0002-0.04 wt %, and bismuth in an amount of about 0.4-0.7 wt %.	Xiao discloses a “magnesium alloy...and further adds elements of Fe, Cu, Ni, and Ag which can enhance the corrosion performance of the magnesium alloy...” Xiao at ¶ 0026, ll. 1-9.  Claim 1 of Xiao discloses a “magnesium alloy, comprising the components at the weight percentages as follows: Al: 13 to 25%, Zn: 2 to 15%; the remainder is Mg, and a sum of the weight percentages of the components is 100%.” Xiao at Claim 1 (emphasis added). Dr. Medlin opines that a POSITA would have understood that the total range of the non-magnesium elements disclosed in Claim 1 of Xiao is 15 to 40%, leaving 60-85 wt% for magnesium. Medlin Decl. Exhibit 2 at ¶ 149. Dr. Medlin also notes that the “Examples of Xiao disclose a range of magnesium from 54.5 wt% - 79.2 wt%.” Id. Thus, Xiao discloses a range of magnesium 54.5-85 wt%, which overlaps and covers much of the claimed range.  This claim element would be anticipated solely by the zirconium concentrations falling within the 0.01-1 and/or 0.01-3 wt% ranges. Claim 14 also requires, in addition to a similar zirconium concentration to the Markush claims, a zinc concentration of 0.1-3 wt%.
<b>Dependent Claim 20</b>	<b>Disclosure of Claim Element in Xiao and/or in Combination with Hassan</b>
20. The magnesium composite as defined in claim	Xiao discloses a “magnesium alloy...and further adds elements of Fe, Cu, Ni, and Ag which can enhance the corrosion performance of the magnesium alloy...” Xiao at ¶ 0026, ll. 1-9.

12, said additive material includes nickel,	<p>Xiao teaches the use of a variety of metals as additive materials. See, e.g., Xiao at ¶ 0026. For instance, Xiao claims that the novel magnesium alloy may “further compris[e] trace elements at the weight percentages as follows: Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%...”Id. at Claim 2. In addition, Example 7 of Xiao particularly discloses intermediate alloys Al-Fe, Al-Ni, Al-Ag, Al-Cu, Al-Zr, Al-Ti and Zinc as additive materials. Id. at ¶ 0060. A POSITA in April 2014 would have understood that all of these intermediate alloys contain metal, including nickel, among other metals. Medlin Decl. Exhibit 2 at ¶ 145. Thus, Xiao discloses a number of metal additives, including nickel, and hence fully discloses all of the limitations of these claim elements.</p>
said nickel constitutes about 0.05-35 wt % of said magnesium composite,	<p>Claim 1 of Xiao discloses: a “magnesium alloy, comprising the components at the weight percentages as follows: Al: 13 to 25%, Zn: 2 to 15%, the remainder is Mg, and a sum of the weight percentages of the components is 100%.” Xiao at Claim 1. And Claim 2 discloses: a “magnesium alloy, according to Claim 1, further comprising the trace elements at the weight percentages as follows: Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%; and a sum of the weight percentages of the components is 100%.” Id. at Claim 2.</p> <p>These metallic elements are found in the additive materials generally disclosed by Xiao (i.e., intermediate alloys: Al-Fe, Al-Ni, Al-Cu, Al-Ti) (Xiao at ¶ 0023) and particularly described in association with Example 7 of Xiao (Id. at ¶¶ 0057–0060) for which Dr. Medlin calculated for Example 7 of Xiao (Medlin Decl. Exhibit 2 at ¶¶ 113–122) the specific weight percentage (ranges) of each additive material or secondary metal.</p>

	<p>In particular, Dr. Medlin determined that the additive materials aluminum-iron with 30 wt% iron, aluminum-nickel with 45 wt% nickel, and aluminum-copper with 40 wt% copper also fall well within the range of about 0.05 wt% to 45 wt% of the mixture. Id. ¶ 122. For example, the aluminum-iron additive constitutes 3.3 wt% of the mixture; the aluminum-nickel additive constitutes 1.1 wt% of the mixture; and the aluminum-copper additive constitutes 0.25 wt% of the mixture. Dr. Medlin expressed his opinion that a POSITA in April 2014 would have also recognized that other intermediate alloy compositions would have fallen within the teachings of Example 7 and other disclosures of Xiao resulting in Al-Fe, Al-Ni, and Al-Cu intermediate alloys constituting about 0.05 wt% to 45 wt% of the mixture. Id. at ¶ 125. Accordingly, this claim element was disclosed by Xiao.</p>
said nickel forms galvanically-active in situ precipitate in said magnesium composite.	<p>Xiao discloses a novel magnesium alloy with high aluminum and zinc content further adding elements Fe, Cu, Ni and Ag which can form intermetallics and enhance the corrosion performance of the magnesium alloy. Xiao at ¶ 0026. Xiao explains the corrosion mechanism within a magnesium alloy in terms of a cathode phase, which Xiao explains includes “the <math>\beta</math> (<math>Mg_{17}Al_{12}</math>) phase and eutectic phase on the grain boundary of the magnesium alloy,” and “the magnesium matrix <math>\alpha</math> phase which functions as an anode phase, so that the matrix and the grain boundary of the magnesium alloy form a large amount of micro-batteries, which [micro-batteries] greatly accelerate the corrosion decomposition of magnesium alloy.” Id.</p> <p>A POSITA in August 2014 would have understood this disclosure of “intermetallic composite micro-particles” to be a</p>

	<p>reference to intermetallic precipitate. Medlin Decl. Exhibit 2 at ¶ 107.</p> <p>A POSITA reading the Xiao reference in April 2014 would have recognized that the precipitation of galvanically-active intermetallic phases (“micro-batteries”) are naturally and necessarily generated by the in situ reaction between magnesium and any and all of the disclosed metal additives forming in situ precipitate. Medlin Decl. Exhibit 2, at ¶ 108. Thus, these claim elements were disclosed by Xiao.</p> <p>These elements are among the claimed “secondary metals” disclosed by Xiao. Thus, all of the limitations of this claim element are disclosed by Xiao.</p>
<b>Dependent Claim 21</b>	<b>Disclosure of Claim Element in Xiao and/or in Combination with Hassan</b>
21. The magnesium composite as defined in claim 20, wherein said additive material includes nickel,	<p>Xiao discloses a “magnesium alloy...and further adds elements of Fe, Cu, Ni, and Ag which can enhance the corrosion performance of the magnesium alloy...” Xiao at ¶ 0026, ll. 1–9.</p> <p>Xiao teaches the use of a variety of metals as additive materials. See, e.g., Xiao at ¶ 0026. For instance, Xiao claims that the novel magnesium alloy may “further compris[e] trace elements at the weight percentages as follows: Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%...” Id. at Claim 2. In addition, Example 7 of Xiao particularly discloses intermediate alloys Al-Fe, Al-Ni, Al-Ag, Al-Cu, Al-Zr, Al-Ti and Zinc as additive materials. Id. at ¶ 0060. A POSITA in April 2014 would have understood that all of these intermediate alloys contain metal, including nickel, among other metals. Medlin Decl. Exhibit 2 at ¶ 145. Thus, Xiao discloses a number</p>

	of metal additives, including nickel, and hence fully discloses all of the limitations of these claim elements.
said nickel constitutes about 3-7 wt % of said magnesium composite.	<p>Claim 1 of Xiao discloses: a “magnesium alloy, comprising the components at the weight percentages as follows: Al: 13 to 25%, Zn: 2 to 15%, the remainder is Mg, and a sum of the weight percentages of the components is 100%.” Xiao at Claim 1. And Claim 2 discloses: a “magnesium alloy, according to Claim 1, further comprising the trace elements at the weight percentages as follows: Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%; and a sum of the weight percentages of the components is 100%.” Id. at Claim 2.</p> <p>These metallic elements are found in the additive materials generally disclosed by Xiao (i.e., intermediate alloys: Al-Fe, Al-Ni, Al-Cu, Al-Ti) (Xiao at ¶ 0023) and particularly described in association with Example 7 of Xiao (Id. at ¶¶ 0057–0060) for which Dr. Medlin calculated for Example 7 of Xiao (Medlin Decl. Exhibit 2 at ¶¶ 113–122) the specific weight percentage (ranges) of each additive material or secondary metal.</p> <p>In particular, Dr. Medlin determined that the additive materials aluminum-iron with 30 wt% iron, aluminum-nickel with 45 wt% nickel, and aluminum-copper with 40 wt% copper also fall well within the range of about 0.05 wt% to 45 wt% of the mixture. Id. ¶ 122. For example, the aluminum-iron additive constitutes 3.3 wt% of the mixture; the aluminum-nickel additive constitutes 1.1 wt% of the mixture; and the aluminum-copper additive constitutes 0.25 wt% of the mixture. Dr. Medlin expressed his opinion that a POSITA in April 2014 would have also recognized that other intermediate alloy compositions could have fallen within the teachings of Example 7 and other</p>

	disclosures of Xiao resulting in Al-Fe, Al-Ni, and Al-Cu intermediate alloys constituting about 0.05 wt% to 45 wt% of the mixture. Id. at ¶ 125. Accordingly, this claim element was disclosed by Xiao.
<b>Dependent Claim 23</b>	<b>Disclosure of Claim Element in Xiao and/or in Combination with Hassan</b>
23. The magnesium composite as defined in claim 12, wherein a dissolution rate of said magnesium composite is at least 45 mg/cm <sup>2</sup> /hr. in 3 wt % KCl water mixture at 90°C. and up to 325 mg/cm <sup>2</sup> /hr. in 3 wt % KCl water mixture at 90°C.	A POSITA in April 2014 would have recognized that Xiao disclosed a table of dissolution rates (“decomposition rates”) because the units of measurement are the units used by POSITAs to measure dissolution rates. Medlin Decl. Exhibit 2 at ¶ 129. The disclosed table provides seven examples (excluding the comparative [prior art] example). Xiao at ¶ 0064. Xiao provides dissolution rates in g/cm <sup>2</sup> /hr, while the claims of the '653 Patent express dissolution rates in mg/cm <sup>2</sup> /hr. A POSITA would have known converting between g/cm <sup>2</sup> /hr to mg/cm <sup>2</sup> /hr, merely requires multiplication by 1,000. Medlin Decl. Exhibit 2 at ¶ 130.  Six of the seven examples (excluding the comparative [prior art] example) provided in Xiao therefore discloses a dissolution rate of “at least 45 mg/cm <sup>2</sup> /hr in 3 wt % KCl water” and “up to 325 mg/cm <sup>2</sup> /hr in 3 wt % KCl water” at 93°C. Given that the dissolution rates of Xiao slightly above (i.e., 3°C above) 90°C are almost all above the claimed floor of “at least 45 mg/cm <sup>2</sup> /hr”. As such and per Dr. Medlin’s opinion, a POSITA in April 2014 would have expected the dissolution rate of the examples in Xiao at 90°C to also be necessarily above the claimed floor of “at least 45 mg/cm <sup>2</sup> /hr.” Medlin Decl. Exhibit 2 at ¶ 205. Thus, this element is disclosed by Xiao.
<b>Dependent Claim 24</b>	<b>Disclosure of Claim Element in Xiao and/or in Combination with Hassan</b>

<p>24. The magnesium composite as defined in claim 12, wherein said additive material is a metal or metal alloy.</p>	<p>Xiao discloses a “magnesium alloy...and further adds elements of Fe, Cu, Ni, and Ag which can enhance the corrosion performance of the magnesium alloy...” Xiao at ¶ 0026, ll. 1–9.</p> <p>Xiao teaches the use of a variety of metals as additive materials. See, e.g., Xiao at ¶ 0026. For instance, Xiao claims that the magnesium alloy may “further compris[e] trace elements at the weight percentages as follows: Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%...” Id. at Claim 2. In addition, Example 7 of Xiao particularly discloses intermediate alloys Al-Fe, Al-Ni, Al-Ag, Al-Cu, Al-Zr, Al-Ti and Zinc as additive materials. Id. at ¶ 0060. A POSITA in April 2014 would have understood that all of these additive materials contain metal(s). Medlin Decl. Exhibit 2 at ¶ 145. Accordingly, Example 7 of Xiao discloses this claim element in accordance with Patent Owner’s definition of “additive material.”</p>
<p><b>Dependent Claim 71</b></p>	<p><b>Disclosure of Claim Element in Xiao and/or in Combination with Hassan</b></p>
<p>71. The magnesium composite as defined in claim 49, wherein at least a portion of said additive material remains unalloyed additive material in said magnesium composite.</p>	<p>Xiao discloses that “[e]lements such as Fe, Cu, Ni, Ag, etc., in the micro-particles.” Id. at ¶ 0026. However, Xiao does not disclose whether any of the additive material remains “unalloyed”.</p> <p>Hassan explicitly discloses that magnesium composites containing additive materials, such as nickel may remain unreacted. In particular, Hassan discloses that the [s]evere reaction between magnesium melt and nickel particulates during DMD processing led to the reduction of the particulate size and formation of Mg<sub>2</sub>Ni intermetallics [13]. The results of quantitative determination of unreacted nickel (see Table I), microstructural characterization illustrating the presence of</p>

	<p>reaction products (see Fig. 1) and XRD results (see Table II) showing the presence of Mg<sub>2</sub>Ni supports the experimental observations.”</p> <p>Ex. 1007 at 2472. Thus, Hassan specifically provides experimental data showing—under similar conditions of magnesium composite fabrication as those in Xiao—that some of the additives, particularly nickel, will remain unreacted and unalloyed.</p> <p>Given the motivation to combine Xiao with Hassan (discussed, <i>supra</i>), Dr. Medlin opined that a POSITA in April 2014 would have combined these two references thereby providing further rationale that a POSITA in April 2014 would find Claim 12 of the ‘653 Patent obvious.</p>
--	---

## X. CONCLUSION

Requester respectfully requests the *ex parte* reexamination of claims 1–5, 7–9, 11–16, 18–21, 23–50, 52–62, 64, 66, 67, and 69–78.

Respectfully submitted,

Date: July 6, 2021

By: \_\_\_\_\_ /Marc A. Brockhaus/  
Marc A. Brockhaus, Reg. No. 40,923  
Dunlap Codding, P.C.  
P.O. Box 16370  
Oklahoma City, OK 73113  
Telephone: (405) 607-8600  
Facsimile: (405) 607-8686

**CERTIFICATION OF SERVICE**

The undersigned certifies that, on July 6, 2021, complete copies of this Request for *Ex Parte* Reexamination, including this Certificate of Service, and all References were served on Patent Owner by First Class U.S. Mail to the following correspondence address of record:

FAY SHARPE LLP  
1228 Euclid Ave., 5<sup>th</sup> Floor  
The Halle Building  
Cleveland, OH 44115

Brian E. Turung, Esq.  
ULMER BERNE  
1660 W. 2<sup>nd</sup> St., Ste. 1100  
Cleveland, OH 44113

Date: July 6, 2021

By: /Marc A. Brockhaus/  
Marc A. Brockhaus, Reg. No. 40,923  
Dunlap Codding, P.C.  
P.O. Box 16370  
Oklahoma City, OK 73113  
Telephone: (405) 607-8600  
Facsimile: (405) 607-8686

Attorney Docket No. 2189.006

Patent 10,329,653

**IN THE UNITED STATES PATENT AND TRADEMARK OFFICE**

In re Patent of:  
Doud, et al.

Attorney Docket No.  
2189.006

U.S. Patent. No.: 10,329,653

Issued: June 25, 2019

Assignee: Terves, LLC

Mail Stop Ex Parte Reexam  
Central Reexamination Unit Commissioner for Patents  
United States Patent and Trademark Office P.O. Box 1450  
Alexandria, VA 22313-1450

---

**DECLARATION OF DANA J. MEDLIN, PH.D., P.E., FASM UNDER 37 C.F.R. § 1.68 IN  
SUPPORT OF REQUEST FOR EX PARTE REEXAMINATION OF U.S. PATENT NO.**

**10,329,653**

Attorney Docket No. 2189.006

Patent 10,329,653

## **I. INTRODUCTION AND SUMMARY OF OPINIONS**

1. My name is Dana J. Medlin. I submit this declaration on behalf of Petitioner Ecometal, Inc., which I understand is challenging the validity of claims 1–5, 7–9, 11–16, 18–21, 23–50, 52–62, 64, 66, 67, and 69–76 (Challenged Claims) of U.S. Patent No. 10,329,653 (the '653 patent '653 Patent) in a request for *ex parte* reexamination before the United States Patent and Trademark Office.

2. I have been asked to provide an opinion on the validity of the Challenged Claims. I previously submitted a substantially similar declaration in an *inter partes* review proceeding, IPR2020-1620, challenging the validity of some of the Challenged Claims. While my analysis there and here does not address every single one of the Challenged Claims, my analysis, below, covers each limitation of all of the Challenged Claims.

3. My opinions are based, at least in part, on the following references, as well as my understanding as to the general knowledge of a person of skill in the art at the time of the provisional application (i.e. April 18, 2014) to which the '653 Patent claims priority:

<u>REFERENCE</u>	<u>DATE OF PUBLIC AVAILABILITY</u>
A Certified English Language Translation of the Chinese Pat. Pub. No. CN 103343271 (Xiao) (Xiao)	October 9, 2013
Hassan, S.F., Gupta, M. Development of high strength magnesium based composites using elemental nickel particulates as reinforcement. <i>Journal of Materials Science</i> 37, 2467–2474 (2002). (Hassan)	June 2002

4. As detailed below, I conclude that the Challenged Claims are either anticipated by Xiao and/or obvious over the combination of Xiao and Hassan.

Attorney Docket No. 2189.006

Patent 10,329,653

5. With respect to the Challenged Claims, and as I discuss further in the following three paragraphs, each of the claims appears to be a reorganization of several basic claim limitations.

6. In particular, each of the Challenged Claims of the ‘653 patent claim a magnesium [alloy] composite<sup>1</sup> that includes *in situ* precipitates of galvanically-active intermetallic phase(s)/particle(s) that allegedly enable “controlled dissolution”<sup>2</sup> of the magnesium [alloy] composite<sup>3</sup> -- i.e., at “a dissolution rate of at least<sup>4</sup> 5 mg/cm<sup>2</sup>/hr. in 3 wt.% KCl water mixture at 90°C.”<sup>5</sup>

7. In each of the Challenged Claims, the magnesium [alloy] composite comprises:

- A. magnesium (Mg) or a magnesium alloy: and
- B. an “additive material”<sup>6</sup> or “secondary metal.”<sup>7</sup>

With respect to this “B” limitation, I collectively consider it an “added metal” limitation because in all of the Challenged Claims reciting an “additive material” each claim further requires that the

---

<sup>1</sup> Challenged Claims **25** (bolded claim numbers indicate an independent claim), **29, 33, 37, 41, 45, 70, 73** and **74** explicitly note that the magnesium [alloy] composite is for use in oil well drilling tool components (e.g. frac balls).

<sup>2</sup> The claimed property of “controlled dissolution” is also referred to in some Challenged Claims as “corrosion.”

<sup>3</sup> For example, this limitation, in particular, is represented by the language “*in situ* precipitation of galvanically-active intermetallic phases to enable controlled dissolution of said magnesium composite” in Challenged Claims **1, 4, 5, 7, 8, 11, 12, 13, 49, 55, 69, 70, 71, 73** and **74** and by the language “a secondary metal to form a galvanically-active intermetallic particle that promotes corrosion of said dissolvable magnesium alloy composite” in Challenged Claims **25, 29, 33, 37, 41, 43,** and **45.** Challenged Claim **1** also explicitly states the “additive material forming precipitant in said magnesium composite.”

<sup>4</sup> Challenged Claims **49** and **73** actually recite “at least at least [sic] 5 mg/cm<sup>2</sup>/hr.” which I have interpreted to mean simply “at least 5 mg/cm<sup>2</sup>/hr.”

<sup>5</sup> Instead of “at least 5 mg/cm<sup>2</sup>/hr.” the value recited in Challenged Claim **74** is “84-325 mg/cm<sup>2</sup>/hr” using the same KCl water mixture at 90°C.

<sup>6</sup> Challenged Claims **1, 4, 5, 7, 8, 11, 12, 13, 49, 55, 69, 70, 71, 73, and 74.**

<sup>7</sup> Challenged Claims **25, 29, 33, 37, 41, 43, and 45.**

Attorney Docket No. 2189.006

Patent 10,329,653

“additive material includes one or more metals selected from the group consisting of” copper, nickel, and cobalt. In most of the Challenged Claims iron is part of the “one or more metals,” in some claims titanium is included, and in one claim silicon is included.<sup>8</sup> The Challenged Claims reciting the “secondary metal” similarly require that the “secondary metal [includes] one or more metals selected from the group consisting of copper, nickel, [and] cobalt” with Challenged Claims 25, 29, 33, and 37 also reciting “titanium and iron” among their group’s members.

8. The claimed magnesium [alloy] composites contain the following explicit weight percentage composition limitations for the:

- magnesium
  - “[greater than/over] 50wt%”
  - “at least 85 wt%”
  - “60-95 wt%”
- “added metal”
  - “additive material constituting about 0.05 wt.%-45 wt.% of” the mixture/composite
  - “additive material includes...said [additive material] constitutes about 0.05 wt.%-35 wt.% of” said magnesium composite
  - “about 0.05-45 wt.% of a secondary metal”
  - “additive material constituting about [0.05/0.1] wt.% of said mixture”<sup>9</sup>
- magnesium alloying elements
  - “one or more metals selected from the group consisting of 0.5-10wt.% aluminum, 0.05-6wt.% zinc, 0.01-3wt.% zirconium, and 0.15-2wt.% manganese”
  - “0.01-1wt.% zirconium”
  - “0.5-10wt.% aluminum; 0.05-6wt.% zinc; 0.15-2wt.% manganese”
  - “0.05-6wt.% zinc; 0.01-1wt.% zirconium”

---

<sup>8</sup> A few of the Challenged Claims also include one or more limitations directed to the melting point of the additive material: “said additive material having a greater melting point temperature than a solidus temperature of said magnesium” (Challenged Claims 1, 12, and 13) and “said additive material having a melting point temperature that is 100°C. greater than a melting temperature of said magnesium or magnesium alloy” (Challenged Claims 12 and 69).

<sup>9</sup> Challenged Claims 1, 12, 49, 73 and 74 specify that the “mixture” is “a mixture of magnesium or a magnesium alloy and an additive material.”

Attorney Docket No. 2189.006

Patent 10,329,653

- “one or more metals selected from the group consisting of 0.5-10wt.% aluminum, 0.1-2wt.% zinc, 0.01-1wt.% zirconium, and 0.15-2wt.% manganese”
- “one or more metals selected from the group consisting of 0.1-3wt.% zinc, 0.01-1wt.% zirconium, 0.05-1wt.% manganese, 0.0002-0.04wt.% boron, and 0.4-0.7wt.% bismuth”

Some of the Challenged Claims expressly require that some of the additive material (or a portion of the additive material<sup>10</sup>) remains unalloyed and this unalloyed material “forms [the] galvanically-active in situ precipitate in said magnesium composite.”<sup>11</sup> The Challenged Claims (73 and 74) allege that the galvanically-active in situ precipitate provides the claimed improved dissolution/corrosion properties of the magnesium [alloy] composite. Two of the Challenged Claims (49 and 73) explicitly attribute the alleged improved dissolution/corrosion properties of the composition to the “additive material located in sufficient quantities in said galvanically-active intermetallic phases so as to obtain a composition and morphology of said galvanically-active intermetallic phases such that a galvanic corrosion rate along said galvanically-active intermetallic phases.”

9. With respect to my opinion that certain Challenged Claims are obvious over the combination of Xiao in view of Hassan, I believe that it would have been obvious for a person having ordinary skill in the art in April 2014 to combine the described and disclosed alloying mixtures, melting practices, and thermal-mechanical processing techniques described by Hassan

---

<sup>10</sup> For example, Challenged Claims 4 and 43 require “wherein said [additive material/secondary metal] includes nickel, . . . said nickel forms galvanically-active in situ precipitate in said magnesium composite.” Challenged Claim 3 presents similarly with copper.

<sup>11</sup> “. . . at least a portion of said additive material remaining **unalloyed additive material**, said magnesium composite including in situ precipitation of **galvanically-active intermetallic phases that includes said unalloyed additive material**” (Challenged Claim 12 (emphasis added); see also Challenged Claim 71); “said additive material located in sufficient quantities in said galvanically-active intermetallic phases so as to obtain a composition and morphology of said galvanically-active intermetallic phases such that a galvanic corrosion rate along said galvanically-active intermetallic phases causes [the claimed dissolution rate]” (Challenged Claim 49).

Attorney Docket No. 2189.006

Patent 10,329,653

and the similar alloying mixtures, melting practices, and thermal-mechanical processing techniques described and disclosed by Xiao to form galvanically-active particles in a magnesium alloy for enhanced dissolution by controlled corrosion processes.

**II. QUALIFICATION AND BACKGROUND**

10. I have over 30 years of experience leading multidisciplinary failure analysis and design projects in metallurgical and materials science engineering, corrosion engineering, materials joining and welding, and biomedical engineering for a variety of companies and firms. My qualifications are detailed in my *curriculum vitae*, attached hereto as Ex. A.

11. In 1988, I earned a B.S. in Mechanical Engineering (Metallurgy Option) from the University of Nebraska at Lincoln. In 1990, I received an M.S. in Mechanical Engineering (Metallurgy Option) from University of Nebraska at Lincoln. In 1993, I earned a Ph.D. in Materials Science Engineering by the University of Nebraska at Lincoln. My doctoral dissertation was entitled "Metallurgical Phase Transformations in the Case Layer of a Carburized Bearing Steel Due to Rolling Contact Fatigue."

12. From 1993 to 1997, I served as a Research Assistant Professor at the Colorado School of Mines, in Golden, Colorado, where I conducted research, taught graduate and undergraduate courses in metallurgical and materials science engineering, supervised and managed graduate and undergraduate student research projects, presented and published technical research results, and conducted failure analysis, engineering design and maintenance metallurgy for several companies and firms.

13. From 1997 to 1998, I worked as a Materials Specialist and Supervisor of the Analytical Services Group at LTV Steel Company, a producer of flat-rolled and coated sheet steels. In that capacity, I supervised personnel and analytical research equipment including a complete

Attorney Docket No. 2189.006

Patent 10,329,653

metallurgical laboratory, two scanning electron microscopes, FTIR, x-ray diffraction, laser surface profilometer, numerous hardness testers, and corrosion testing. I also evaluated metallurgy quality problems with sheet steel and coatings, conducted mechanical property and fatigue evaluations, performed corrosion testing and failure analysis.

14. From 1998 to 2000, I was a Specialist and Principal Engineer for the Timken Company, an alloy steel and bearing manufacturing company. In those roles, I participated in the microstructural design of debris resistant bearings and heat treatment processing, performed basic static and dynamic mechanical testing on standard test samples and prototype components, managed all metallurgical and heat-treat quality issues, supervised validations and documentation for production derivatives, and recommended potential design options. I was also involved with failure analysis of returned products and issues involving potential product liability litigation.

15. From 2000 to 2005, I was the Global Metallurgy and Materials Leader and Principal Engineer for Zimmer, Inc., a manufacturer of orthopedic, spinal, and dental medical devices. I coordinated materials research efforts between branches of the company, including eliminating research redundancies, resolving systemic manufacturing issues, prioritizing research projects, and developing best practice recommendations. I was the team leader and obtained three patents in developing a process for metallurgically attaching trabecular metal (tantalum foam) to titanium substrates. I was a key member of the project team that was responsible for metallurgically attaching trabecular metal coating to new products. I managed the corrosion laboratory, where I performed and directed corrosion tests and analysis including long-term immersion testing and potentiodynamic anodic polarization (PAP) testing. I was also the project manager for the development of a surface carburization heat treatment for austenitic stainless-steel trauma products. Further, I designed test protocols for new product static and dynamic testing, including

Attorney Docket No. 2189.006

Patent 10,329,653

bending fatigue, uniaxial fatigue, shear, tensile, fracture toughness, coating adhesion, corrosion testing, residual stress, chemical analysis, and microstructural characterization.

16. From 2005 to 2011, I was the NUCOR Professor of Metallurgy at the South Dakota School of Mines and Technology. In that capacity, I taught graduate and undergraduate courses in materials and metallurgical engineering and developed a research program in physical and mechanical metallurgy. From 2010 to 2011, served as the Director of the Biomedical Engineering Graduate Program.

17. Also from 2005 to 2011, I formed Medlin Engineering Services and served as a private engineering consultant, where I conducted forensic engineering investigations, failure analysis, undertook engineering design, and maintenance metallurgical engineering problems for several companies and firms.

18. From 2011 to 2015, I was a senior managing consultant for Engineering Systems, Inc., a consulting engineering and investigation company in Omaha, Nebraska. I conducted forensic engineering investigations, failure analysis, engineering design, and provided expert testimony and maintenance metallurgical engineering problem solving for a number of companies and firms.

19. Also, from 2011 to 2015, I served as an adjunct professor at the South Dakota School of Mines and Technology where I provided graduate student guidance and served as a graduate student committee member and an advisor in the Metallurgical Engineering and Biomedical Engineering program.

20. From 2015 to May 2021, I was a senior managing consultant for EAG Laboratories, where I have conducted engineering investigations, failure analysis, engineering design, expert testimony and maintenance metallurgical engineering problem solving, and served as a technical

Attorney Docket No. 2189.006

Patent 10,329,653

expert in litigation involving failure analysis, materials and metallurgical engineering, biomedical engineering, corrosion engineering, design and safety issues, and manufacturing problems. In June 2021 I became the Vice President for EAG Laboratories where I continue to conduct engineering investigations, failure analysis, engineering design, expert testimony and maintenance metallurgical engineering problem solving, and served as a technical expert in litigation involving failure analysis, materials and metallurgical engineering, biomedical engineering, corrosion engineering, design and safety issues, and manufacturing problems. In addition, I also have responsibility for managing the failure analysis business and personnel aspects for the company.

21. From 2019 to May 2021, I was the Kielhorn Professor in the Department of Materials Joining & Welding Engineering at LeTourneau University in Longview, Texas. In that role, I taught graduate and undergraduate courses in materials and metallurgical engineering, materials and joining welding engineering, as well as developed a research program in welding related metallurgy.

22. I have authored or co-authored more than 40 peer-reviewed papers in materials and metallurgical engineering. I have given more than 100 presentations. I am a Licensed Professional Engineer in Ohio, Nebraska, and Texas. I am a member of the American Society for Materials International; Failure Analysis Society, ASM International; International Metallographic Society; American Society for Testing Materials; The Metallurgical Society; the Professional Engineering Examination Board; the Professional Engineering Examination Review Committee; Society for Biomaterials; Society for Automotive Engineers; the Iron & Steel Society; and the National Association of Corrosion Engineers. I have been honored as a Fellow of Alpha Sigma Mu, an International Professional Honor Society for Materials Science and Engineering; awarded tenure

Attorney Docket No. 2189.006

Patent 10,329,653

and professorship at the South Dakota School of Mines & Technology; honored as the NUCOR Professor at the South Dakota School of Mines & Technology; and honored as a fellow of ASM.

23. I am a named inventor on five U.S. patents and two U.S. patent applications. Those patents and patent applications are listed in my attached C.V.

**III. UNDERSTANDING OF PATENT LAW**

24. I am not an attorney. I have been provided with an understanding of the patent law relevant to conducting the analysis given in this report. The following represents a summary of my understanding of these issues.

25. I understand the patent-at-issue is the '653 Patent and that prior art to the '653 Patent includes patents and printed publications in the relevant art that predate the alleged priority date of the '653 Patent, which I have been told to assume is April 18, 2014.

26. I understand that a patent claim is “anticipated,” and thus invalid, if each and every element of that claim is disclosed expressly, implicitly, and/or inherently in a single prior art reference, arranged in the prior art reference as arranged in the claim. I have also been told that even if a reference does not expressly spell out all the limitations arranged or combined as in the claim, the reference may still anticipate the claim if a person having ordinary skill in the art (POSITA), reading the reference, would “at once envisage” the claimed arrangement or combination.

27. With respect to claimed compounds or compositions, I understand that anticipation only requires disclosure of the claimed compound or composition, not its properties or use. In other words, if the claimed composition is identical to a composition disclosed by a prior art reference, it does not matter for purposes of anticipation that the Patentee recognized additional properties of that composition.

Attorney Docket No. 2189.006

Patent 10,329,653

28. I also understand that when, as by a recitation of ranges or otherwise, a claim covers several compositions, the claim is anticipated if even just one of them is in the prior art. I have also been told that when the prior art discloses a range of values which touches or overlaps the claimed range, even if no specific examples falling within the claimed range are disclosed in the prior art reference, that reference still anticipates the claim if the claimed subject matter is disclosed in the reference with "sufficient specificity."

29. I further understand for a disclosure to be implicit or inherent it must be a necessary feature or result of an embodiment sufficiently described and enabled in the prior art reference. In other words, that a certain result or characteristic ***may occur or be present*** in the prior art reference is not enough; that a certain result or characteristic must always occur (or be present) for the result or characteristic to be implicit or inherently disclosed. However, it is not necessary that a POSITA would have recognized that implicit or inherent disclosure at the time of invention.

30. I understand that a patent claim is invalid as "obvious" if the differences between the prior art and the claimed invention are such that they would have been "obvious" to a POSITA before the effective filing date of the claimed invention (i.e., April 18, 2014).

31. I also understand that a patent claim can be obvious in light of a single prior art reference if it would have been obvious to modify that single reference to arrive at the patented invention.

32. I have been further informed that obviousness does not require that a single reference disclose all of the requirements of the claim. Rather, two or more references can be combined if a POSITA would have combined such references.

33. In reaching a conclusion on whether references would have been combined by POSITAs at the time of the invention, I have been informed that I may consider, among other

Attorney Docket No. 2189.006

Patent 10,329,653

things: 1) whether the change was merely the predictable result of using prior art elements according to their own functions; 2) whether there is some teaching or suggestion in the prior art to make the modification or combination of elements claimed in the patent; 3) whether the innovation applies unknown technique that had been used to improve a similar device or method in a similar way; and 4) whether the claimed invention would have been obvious to try, meaning that the claimed and innovation was one of a relatively small number of possible approaches to the problem with a reasonable expectation of success by those having ordinary skill in the art.

34. I further understand if a POSITA can implement a predictable variation, obviousness bars its patentability. For the same reason, if a technique has been used to improve one device and a POSITA would recognize that it would improve similar devices in the same way, using the technique is obvious.

35. I understand that a claim may be obvious if common sense directs one to combine multiple prior art references or add missing features to reproduce the alleged invention recited in the claims.

36. Furthermore, I also have been informed that factors, such as a long felt need for the solution provided by the claimed invention, unsuccessful attempts by others to find the solution provided by the claimed invention, unexpected and superior results from the claimed invention, and other evidence tending to show obviousness or nonobviousness, are a consideration.

37. I understand that a POSITA is determined by considering: 1) the education level of the inventor; 2) the type of problems encountered in the art; 3) prior art solutions to these problems; 4) the rapidity with which innovations are made; 5) the sophistication of the technology; and 6) education level of active workers in the field.

Attorney Docket No. 2189.006

Patent 10,329,653

38. Based on these factors and my review of the '653 Patent (as set forth below) I have determined that a POSITA at the time of invention of the '653 Patent (i.e. April 18, 2014), would have had at least a bachelor's degree in metallurgical, materials, or mechanical engineering, or the equivalent, and at least five years of experience in metallurgical and materials engineering, or an equivalent amount of relevant work and/or educational experience.

39. Based on my level of education and experience, I am familiar with the level of knowledge that a POSITA would have possessed at the time of the invention, i.e., April 18, 2014, and by then, I was a POSITA.

**IV. DESCRIPTION OF THE TECHNOLOGY AND OVERVIEW OF THE '653 PATENT**

40. The '653 Patent ('653 Patent) is entitled "Galvanically-Active in Situ Formed Particles for Controlled Rate Dissolving Tools."

41. The application for the '653 Patent (U.S. Patent Application No. 15/641,439) was filed July 5, 2017. '653 Patent, front page.

42. I understand the '653 Patent is a "division" of the patent application that became U.S. Patent No. 9,903,010, which was filed on April 17, 2015. '653 Patent, second page.

43. I also understand that the '653 Patent relates to U.S. Provisional Patent Application No. 61/981,425, filed on April 18, 2014. '653 Patent, second page.

44. Therefore, I have been told that the '653 Patent has a "priority date" of April 18, 2014.

45. In its "Background of the Invention," the '653 Patent notes that the "ability to control the dissolution of a down hole [oil] well component in a variety of solutions is very important to the utilization of non-drillable completion tools, such as sleeves, frac balls, hydraulic

Attorney Docket No. 2189.006

Patent 10,329,653

actuating tooling, and the like.” ‘653 Patent at 1:16–21. And that “[r]eactive materials for this application, which dissolve or corrode when exposed to acid, salt, and/or other wellbore conditions, have been proposed for some time. Generally, these components consist of materials that are engineered to dissolve or corrode.” *Id.* at 1:21–26.

46. The ‘653 Patent continues on to describe alleged problems with the prior art, as follows:

While the prior art well drill components have enjoyed modest success in reducing well completion costs, their consistency and ability to specifically control dissolution rates in specific solutions, as well as other drawbacks such as limited strength and poor reliability, have impacted their ubiquitous adoption.

‘653 Patent at 1:31–36.

47. The ‘653 Patent then provides an overall direction of its solution to these alleged problems noting that:

Ideally, these components would be manufactured by a process that is low cost, scalable, and produces a controlled corrosion rate having similar or increased strength as compared to traditional engineering alloys such as aluminum, magnesium, and iron.

‘653 Patent at 1:36–40.

#### A. Basic Knowledge of a POSITA in April 2014

48. Having read the ‘653 Patent and the references discussed below, I believe the following information is useful to explain what certain disclosures would have meant to a POSITA (with respect to the subject matter of this patent in April 2014) and how a POSITA would have understood the terminology and basic chemical/materials/metallurgical scientific concepts underlying the ‘653 Patent and the references relied on below:

49. A POSTIA would have understood in April 2014, that an “alloy” is a metal made by combining two or more metallic elements (e.g., combinations of Au (gold) and Ag (silver)).

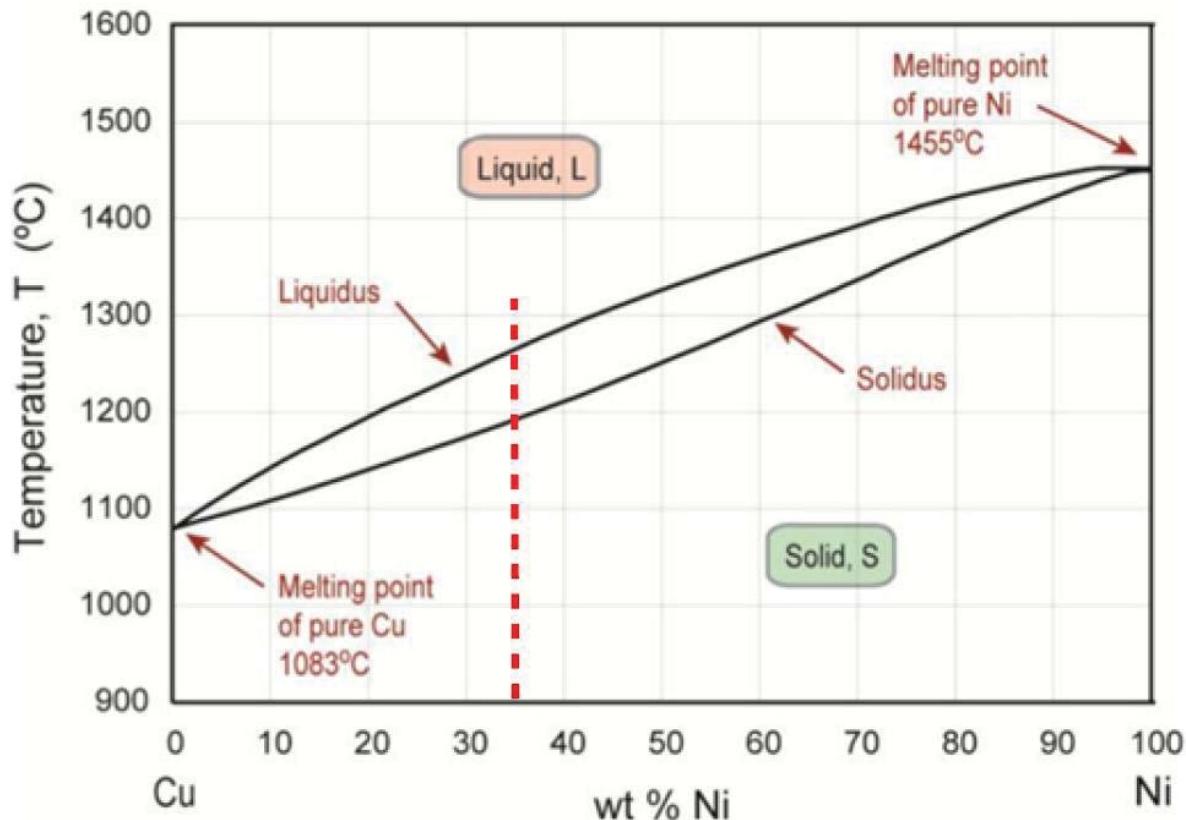
Attorney Docket No. 2189.006

Patent 10,329,653

The physical properties of an alloy are determined in great part by the phases present, and those phases depend on the alloy composition and the thermal and mechanical treatment to which the alloy has been exposed.

50. A POSITA in (and well-before) April 2014 would have routinely referenced “phase diagrams” to reliably predict under what conditions phase changes will occur in an alloy system, even the brief description of FIG. 4 in the ‘653 Patent acknowledged that phase diagrams are “typical.” ‘653 Patent at 15:49. In particular, a “phase diagram” is a graphical representation of the phases present in a particular alloy determined by experimental observations under selected conditions of temperature, pressure, and composition when the system is in thermodynamic equilibrium, and thus provides fundamental physical information about the equilibrium microstructures present in the alloy. Within an alloy system, POSITAs would have understood in (and well-before) April 2014 that one, two, or more different phases can be present at the same time. In a simple two-component, or binary system, where the components have total solubility in each other, the components form a single “solid solution.” For these systems, the phase diagram is especially simple, since only one phase exists in the solid state.

51. The following graph (taken from “Teach Yourself Phase Diagrams and Phase Transformations” Professor Mike Ashby, 5th Edition, March 2009 Cambridge) is an illustrative phase diagram for copper-nickel (aka cupronickel) alloys that vary in only two dimensions: temperature and alloy composition. With respect to the alloy composition, the x-axis begins at the left with pure copper (Cu) and ends on the right with pure nickel (Ni). This illustrative phase diagram for Cu-Ni shows three phases: liquid, solid, and slurry (i.e., a mixture of solid and liquid).

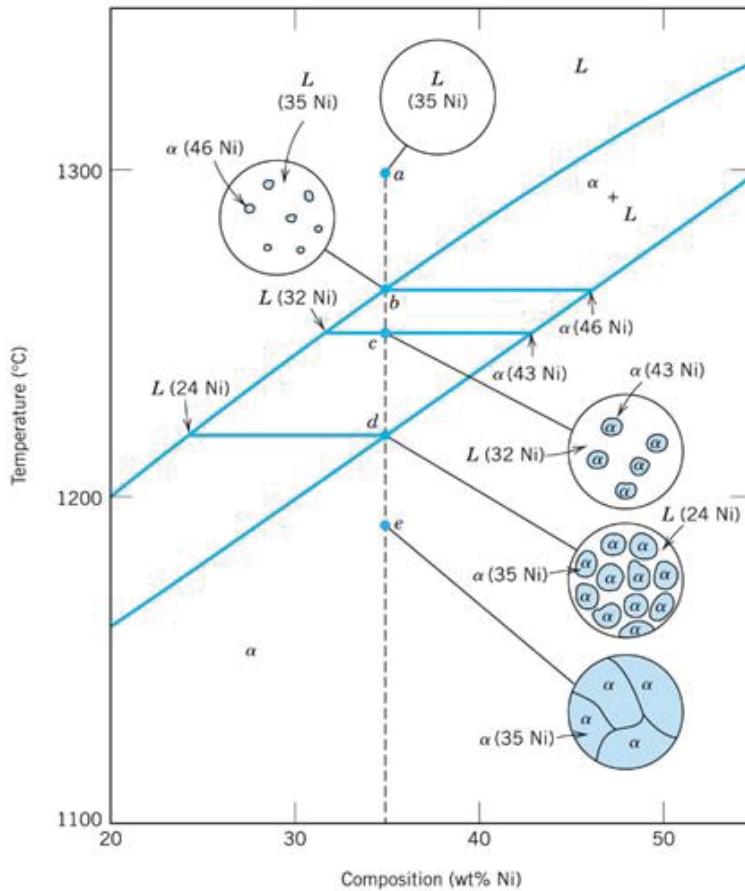


Above the “Liquidus” curve in a phase diagram, all of the alloy is in the molten or liquid phase or state. Below the “Solidus” curve in a phase diagram, all of the alloy is in the solid phase or state. (The liquid state is also sometimes referred to by POSITAs as being a “melt” and the process of changing from a solid state into a liquid state is referred to by POSITAs as “melting.”) For example, based on this phase diagram a POSITA in (and well-before) April 2014 would have understood that a mixture of 35 wt% Ni and 65 wt% Cu at 1300°C is entirely liquid at equilibrium, even though Ni by itself does not melt until 1455°C. The POSITAs would have similarly understood if 35 kg of solid Ni at 1300°C is added to 65 kg of molten Cu at 1300°C, the 35 kg of nickel will dissolve over a period of time into the already melted Cu forming a homogeneous mixture (once equilibrium is reached) even though the Ni added was not in a molten state.

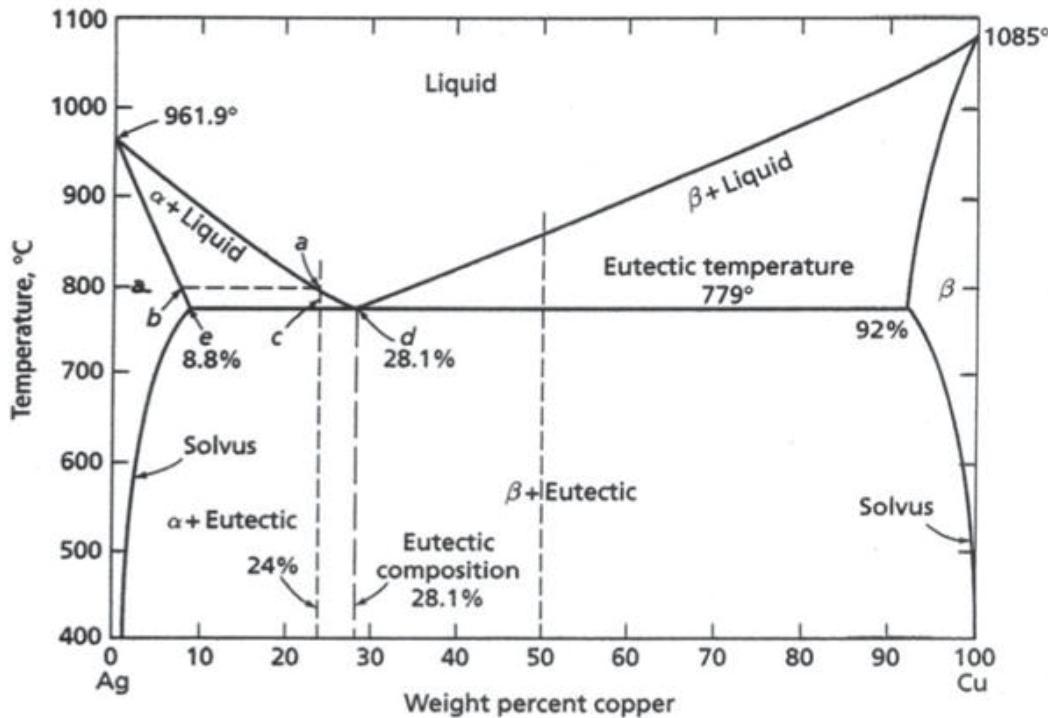
Attorney Docket No. 2189.006

Patent 10,329,653

52. An illustrated portion of the Cu-Ni phase diagram (below) (taken from “Materials Science and Engineering, 6th Edition by Callister and Rethwisch, 2003) shows only a limited range of Ni from 20% to 55%. If the same 35/65 wt% mixture is then cooled from 1300 °C to about 1240°C, a portion of the mixture begins to precipitate out of “the melt.” As would have been understood by a POSITA using this phase diagram, the initial precipitate formed contains about 46 wt% Ni (i.e., the point where the solidus curve intersects a horizontal line from the (35% Ni, 1240°C) position on the diagram). Upon further cooling, the composition of material solidifying follows the solidus curve, depleting the melt of nickel (i.e. the liquid phase will contain less nickel while the solid phase contains more nickel). Between 1240°C and about 1215°C, the material is a mixture of molten and solid alloy, and below the solidus curve, the alloy is completely solid.



53. A POSITA in (and well-before) April 2014, would have understood that in a phase diagram for a system having components only partially soluble in the solid state, two solid phases will co-exist, making the phase diagram more complex. One special case of this type of system is called eutectic. “Eutectic” means that when the two components are mixed, the melting point is lower than the melting temperatures of the two components separately. An illustrative example of a eutectic phase diagram is the Ag-Cu binary phase diagram (taken from Taylor, Shannon submitted to the Dept. of Materials Science and Engineering in partial fulfillment of the Requirements for the Degree of B.S. at the Massachusetts Institute of Technology (June 2013), citing McGraw-Hill Book Co. Inc. 1958 *Constitution of Binary Alloys*, Hansen, M. and K. Anderko) is shown below:



54. As would have been understood by a POSITA in (and well-before) April 2014 using the foregoing phase diagram, upon cooling to 800°C, an alloy melt of 24 wt% Cu and 76 wt% Ag begins to solidify as it crosses the liquidus line at point “a.” The initial solid precipitating out has the composition designated on the solidus curve at point “b” and is typically referred to as an “ $\alpha$  phase.” The  $\alpha$  phase--in this phase diagram--is primarily Ag with a small amount of Cu in solid solution. This is analogous to the  $\beta$  phase on the right side of this phase diagram (which constitutes primarily Cu with small amounts of Ag in solid solution). Upon further cooling of the 24 wt% Cu and 76 wt% Ag composition to the eutectic temperature of 779°C, the remaining molten material solidifies as eutectic phase, the eutectic composition indicated by point “d.” At point d, the eutectic reaction  $L \rightarrow \alpha + \beta$  occurs. The resulting solid alloy is, thus, a mixture of  $\alpha$  phase “grains” and eutectic grains wherein the eutectic grains have a lamellar structure of mixed  $\alpha$  and  $\beta$  phases. (To a POSITA in (and well-before) April 2014, “grain” refers to a 3-dimensional crystallite.

Attorney Docket No. 2189.006

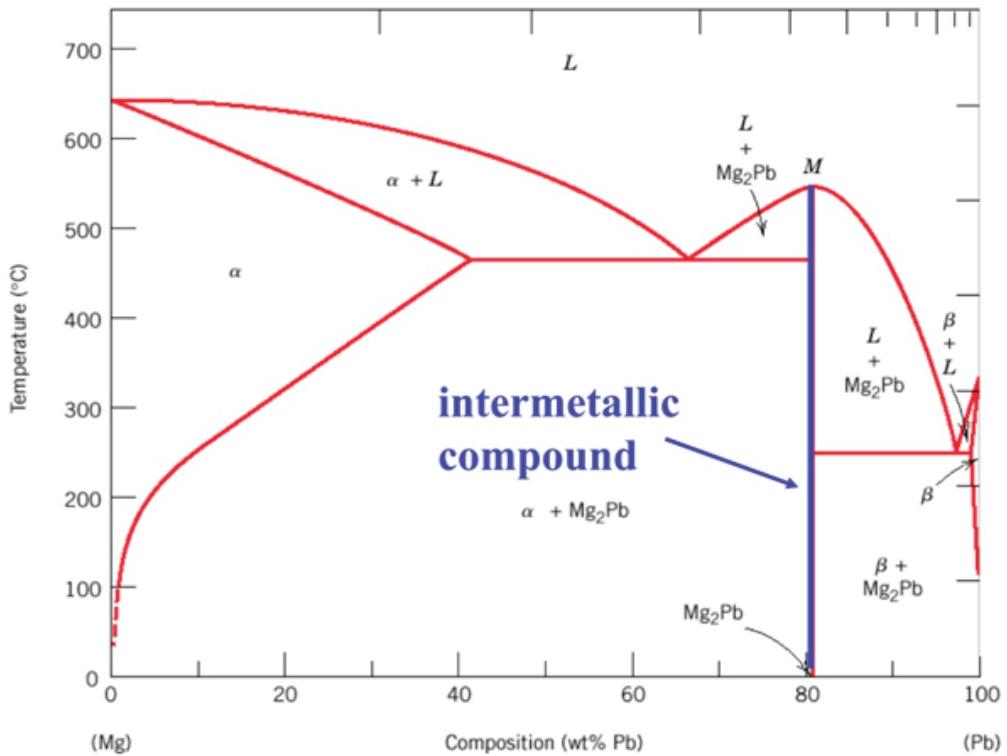
Patent 10,329,653

Grains/crystallites form in alloys during cooling. And the areas where crystallites meet are called a “grain boundary.”)

55. A POSITA in (and well-before) April 2014 would have been aware that many metal alloy systems form intermediate compounds as temperature and other conditions are varied including, for example, CuAl<sub>2</sub>, Al<sub>3</sub>Ni and Fe<sub>3</sub>C. If the components of a precipitated intermediate compound are both metallic, it would have been and still is called an “intermetallic compound” by POSITAs. As a single phase of fixed composition or stoichiometry, intermediate phases have unique melting points and, thus, appear as a vertical line on a phase diagram. For example, the Mg-Pb phase diagram below (taken from MSE 2090: Introduction to Materials Science Chapter 9, Phase Diagrams; adapted from Phase Diagrams of Binary Magnesium Alloys, A.A. Nayeb-Hashemi and J.B. Clark (Editors), 1988 (with annotations added)) can be thought of as two joined eutectic diagrams, one for Mg-Mg<sub>2</sub>Pb and the other for Mg<sub>2</sub>Pb-Pb. The intermetallic compound M (Mg<sub>2</sub>Pb) is a vertical line at approximately 80% Pb on the phase diagram.

Attorney Docket No. 2189.006

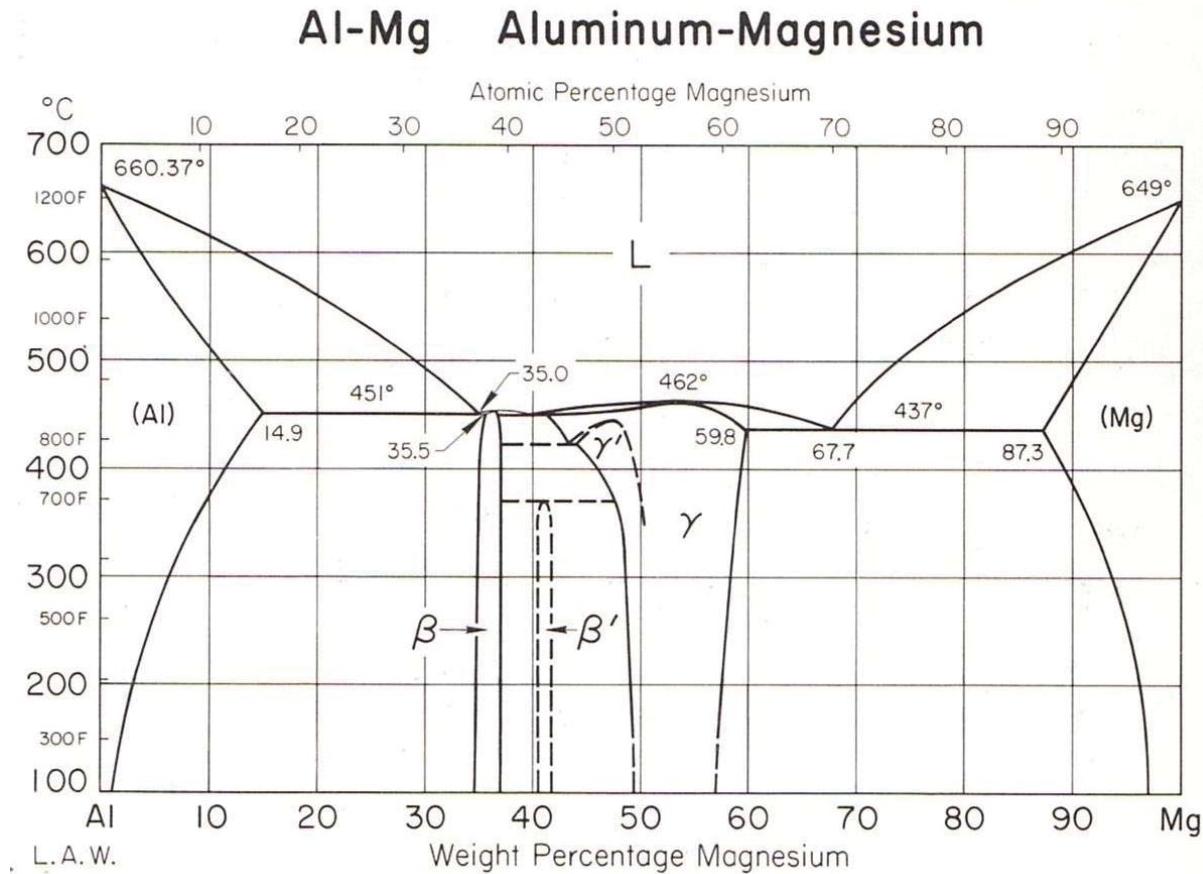
Patent 10,329,653



56. In another example phase diagram for Al-Mg shown below (taken from the ASM Metals Handbook, 8th Edition, Vol. 8, Metallography, Structures and Phase Diagrams, 1973).

Attorney Docket No. 2189.006

Patent 10,329,653



In this example, the (Mg) matrix at the right side of the phase diagram is the same as the  $\alpha$  phase or anode phase mentioned in ¶ 0026 of Xiao Xiao (discussed below). The intermediate  $\gamma$  phase in the above diagram equates to the cathode phase as well as the  $\beta$  phase referred to in ¶ 0026 of Xiao. In this example, the intermediate phases ( $\gamma$  and  $\beta$ ) have very narrow ranges of alloy contents that are near the stoichiometric chemistries which is typical for some phase systems.

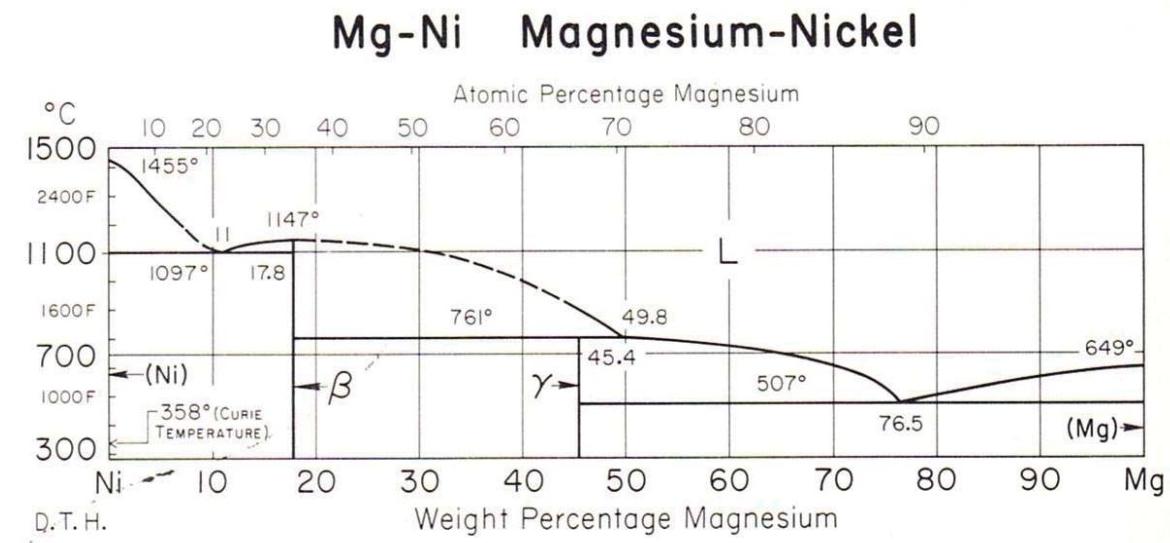
57. A POSITA would have understood in (and well-before) April 2014 that these phase diagrams represent equilibrium conditions. Thus, a POSITA in April 2014 would have understood that additions of higher melting point elements that would completely dissolve (“melt”) in a molten alloy if the mixture was given time to reach equilibrium for the given temperature and composition of the constituents, would also have understood if not given that sufficient time to reach

Attorney Docket No. 2189.006

Patent 10,329,653

equilibrium before the mixture is cooled then the added higher melting point elements would not be completely dissolved prior to solidification of the alloy. Thus, a POSITA in April 2014 would have understood that a controlled cooling rate of the alloy would have limited the extent of dissolution of the higher melting point element in the mixture. Further, a POSITA in April 2014 would have understood that if the higher melting point element were added as particles at a specific wt%, smaller size particles would have greater surface area and would therefore dissolve faster than larger particles.

58. Intermetallic particles -- A POSITA would have already known well before April 2014 that Cu, Ni, and Fe, among other metallic elements, form intermetallic compounds with magnesium. This pre-invention knowledge is evidenced by the diagram below taken from the ASM Metals Handbook, 8th Edition, Vol. 8, Metallography, Structures and Phase Diagrams, 1973.

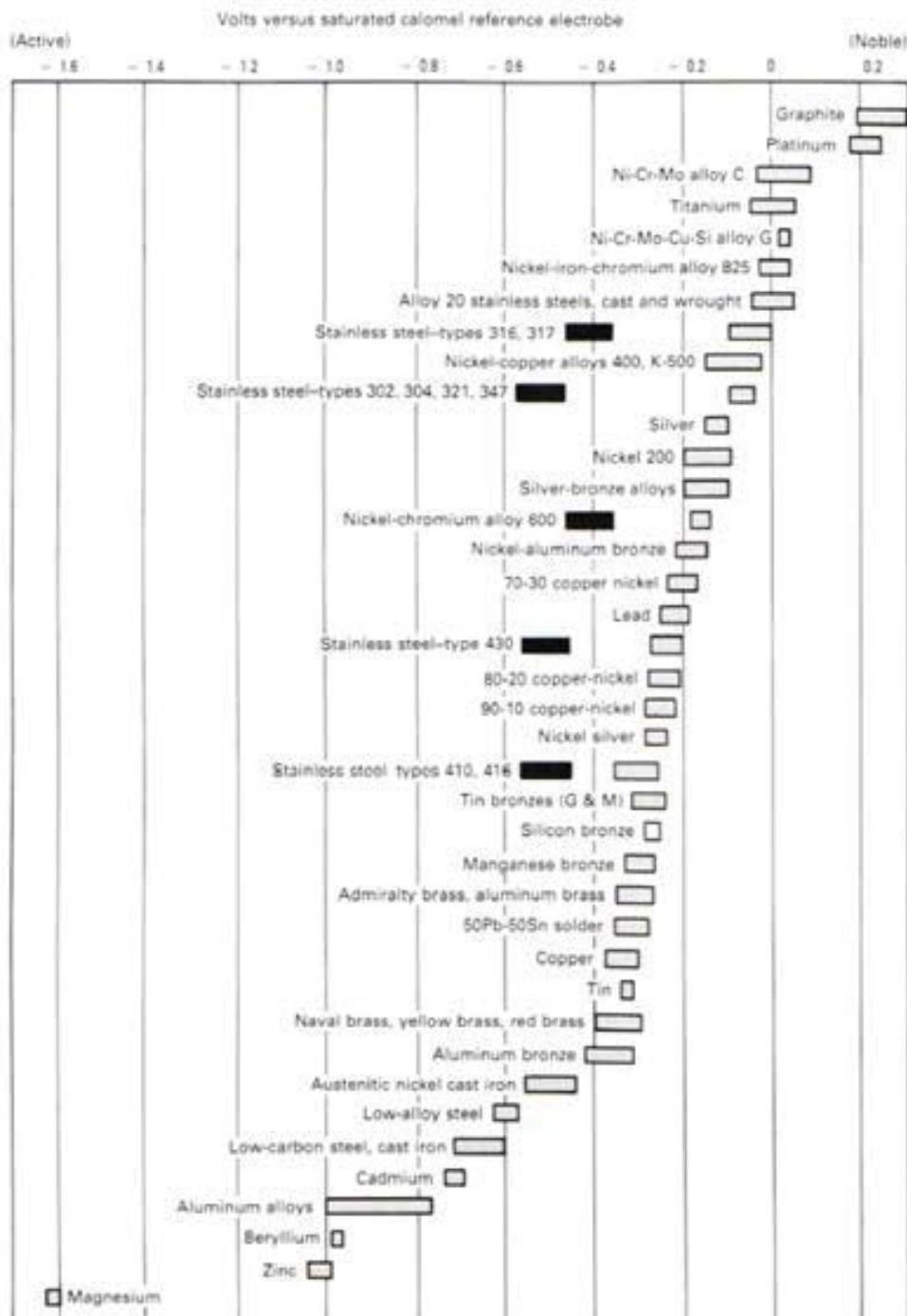


59. Galvanic Corrosion - A POSITA in (and well-before) April 2014 would have known that when two dissimilar metals or alloys are coupled together in a corrosive environment, one of the alloys will preferentially corrode while the other is protected from corrosion. The table

Attorney Docket No. 2189.006

Patent 10,329,653

below is referred to by POSITAs as “the Galvanic Series,” which lists the corrosion potential of common metals and alloys.



Principles and Prevention of Corrosion, 2nd Ed., D.A. Jones, 1996, p 170.

Attorney Docket No. 2189.006

Patent 10,329,653

The table is arranged to illustrate that an alloy or metallic element will preferentially corrode when coupled to another alloy or metallic element with a more positive or noble potential on the Galvanic Series. In other words, when coupled together, the alloy listed lower on the Galvanic Series will corrode and the alloy listed higher on the Galvanic Series will not corrode. In addition, the farther away the coupled alloys are on the list from one another, the higher driving force for corrosion.

60. As shown above, for example, magnesium is at the bottom of the Galvanic Series, and when coupled with other metals that contain elements such as aluminum, copper, zinc, silver, iron, and titanium, the magnesium will corrode, and the other elements will be protected from corrosion. In this sense, the magnesium when combined with other metals acts like a battery with an anode, a cathode, and “an electrolyte.” When the ends of the battery are connected through an electrical circuit, the anode inside the battery will corrode developing an electrical current to energize the circuit. When the anode is fully consumed, the battery is dead.

61. The intermetallics in a magnesium alloy mixture typically act as cathodes and the matrix material typically acts as an anode. When the magnesium alloy is exposed to a conductive solution (such as potassium chloride), the alloy will corrode as discussed above in conjunction with the Galvanic Series. Specific types and amounts of particular intermetallics will be more cathodic to the magnesium than other intermetallics and, thus, will cause the alloy to corrode at faster rates.

62. Alloy Processing - A POSITA in April 2014 would have understood that a key microstructural parameter in an alloy is the grain size, that grain size and shape describe the morphology of a grain, and that the morphology is determined in large part by the mixing prior to solidification and by any post-solidification heat treatment and/or deformation processing. A POSITA in April 2014 would have known that grain size of these alloys can be controlled by solute

Attorney Docket No. 2189.006

Patent 10,329,653

alloying, heat treatments, and forming the metals at ambient and elevated temperatures. With specific heat treatment processing and forming conditions, the grain size of a magnesium alloy can be controlled which in turn changes the mechanical performance of the alloy depending upon the application. Additionally, a POSITA in April 2014 would have understood that the rate of an electrochemical reaction is dependent on surface area which in turn depends on particle or grain size of the reactants. These principles were well known to a POSITA prior to April 2014 and common knowledge in the technical literature. In addition, it was also well known how each alloying element affects the mechanical properties as well as the corrosion resistance of this alloy group.

#### **B. Alleged Invention of the '653 Patent**

63. The '653 Patent's purported solution to its alleged problem<sup>12</sup> is a "magnesium composite for use as a dissolvable component in oil drilling" such as "a ball or other tool component in a well drilling or completion operation such as, but not limited to, a component that is seated in a hydraulic operation that can be dissolved away after use so that no drilling or removal of the component is necessary." '653 Patent at 1:53–59. The '653 Patent describes the purportedly inventive magnesium composite as being used to form a "castable, moldable, or extrudable component." *Id.* at 2:18–20.

64. The purportedly inventive magnesium composite would include "at least 50 wt.% magnesium" with "one or more additives added to a magnesium or magnesium alloy to form the

---

<sup>12</sup> As I already noted above, the '653 Patent described the problem as follows: "While the prior art well drill components have enjoyed modest success in reducing well completion costs, their consistency and ability to specifically control dissolution rates in specific solutions, as well as other drawbacks such as limited strength and poor reliability, have impacted their ubiquitous adoption."

'653 Patent ('653 Patent) at 1:31–36.

Attorney Docket No. 2189.006

Patent 10,329,653

novel magnesium composite.” *Id.* at 2:20–24. The “one or more additives can be selected and used in quantities so that galvanically-active intermetallic or insoluble precipitates form in the magnesium or magnesium alloy while the magnesium or magnesium alloy is in a molten state and/or during the cooling of the melt.” ‘653 Patent *Id.* at 2:24–29.

65. The ‘653 Patent further describes that the “one or more additives can be added to the molten magnesium or magnesium alloy at a temperature that is greater than the melting point of the one or more additives.” *Id.* at 2:53–56. “Generally, the temperature of the molten magnesium or magnesium alloy is at least about 10°C. less than the melting point of the additive added to the molten magnesium or magnesium alloy during the addition and mixing process, typically at least about 100°C. less than the melting point of the additive added to the molten magnesium or magnesium alloy during the addition and mixing process.” *Id.* at 3:2–8. According to the ‘653 Patent, the result of this process is that it “can be used to achieve a specific galvanic corrosion rate in the entire magnesium composite and/or along the grain boundaries of the magnesium composite.” *Id.* at 3:16–18.

66. The ‘653 Patent specifically notes that the “invention adopts a feature that is usually a negative in traditional casting practices wherein a particle is formed during the melt processing that corrodes the alloy when exposed to conductive fields and is imbedded in eutectic phases, the grain boundaries, and/or even within grains with precipitation hardening.” *Id.* at 3:19–24.

67. This purportedly “results in the ability to control where the galvanically-active phases are located in the final casting, as well as the surface area ratio of the *in situ* phase to the matrix phase, which enables the use of lower cathode phase loadings as compared to a powder metallurgical or alloyed composite to achieve the same dissolution rates.” *Id.* at 3:34–30. According to the ‘653 Patent, the *in situ* formed galvanic additives can be used to improve certain

Attorney Docket No. 2189.006

Patent 10,329,653

mechanical properties of the magnesium composite, including ductility, tensile strength, and/or shear strength. ‘653 *Patent Id.* at 3:24–30.

68. The ‘653 Patent further explains that in one non-limiting embodiment the present invention is directed to:

a ball or other tool component in a well drilling or completion operation such as, but not limited to, a component that is seated in a hydraulic operation that can be dissolved away after use so that no drilling or removal of the component is necessary. Tubes, valves, valve components, plugs, frac balls, and other shapes and components can also be formed of the novel magnesium composite of the present invention.

*Id.* at 1:51–59.

69. The ‘653 Patent goes on to describe many “non-limiting aspects” of the alleged invention. For example, the ‘653 Patent explains that the “magnesium composite is used to form a castable, moldable, or extrudable component.” *Id.* at 2:17–18. In this example, the magnesium composite would include at least 50 wt % magnesium with one or more “additives” added to the magnesium or magnesium alloy. *Id.* at 2:17–22. The “additives” are added in a weight percent that is less than the weight percent of the magnesium or magnesium alloy. *Id.* at 2:28–30. The one or more additives are typically added to the magnesium or magnesium alloy while it is in a molten state at a temperature that is less than the melting point of the one or more additives. *Id.* at 2:37–40. During mixing of the one or more additives to the molten magnesium or magnesium alloy, the one or more additives are typically not caused to fully melt. *Id.* at 2:49–53.

70. In forming a composite with the magnesium or magnesium alloy, the one or more additives form “secondary metallic alloys with the magnesium and/or other metals in the magnesium alloy.” *Id.* at 2:56–61. According to the ‘653 Patent, as the molten metal cools, the

Attorney Docket No. 2189.006

Patent 10,329,653

newly formed secondary metallic alloys precipitate out of the molten metal and form the in situ phase to the matrix phase in the cooled and solid magnesium composite. *Id.* at 2:62–65.

71. After mixing, the molten magnesium or magnesium alloy and the one or more additives are cooled to form a solid component. *Id.* at 2:65–3:2. Generally, the temperature of the molten magnesium or magnesium alloy is about 10°C less than the melting point of the one or more additives added during the addition and mixing process. *Id.* at 3:2–13.

72. The '653 Patent refers to the particles that do not melt and/or newly formed “secondary metallic alloys” as “in situ particle formation.” *Id.* at 3:13–15.

### C. Aspects of the Prosecution History of the '653 Patent Relevant to my Opinions

73. The application that became the '653 Patent was filed on July 5, 2017. '653 Prosecution History, at pp. 1306–55.

74. On September 20, 2017, Patent Owner disclosed to the Patent Office twenty-six (26) U.S. Patents, thirty-four (34) U.S. Patent Publications, fifteen (15) Foreign Patent Documents, and thirteen (13) Non-Patent Literature Documents. *Id.* at 864–872. The fifteen (15) Foreign Patent Documents included Chinese Patent Publication No. CN103343271A (in Chinese) accompanied by an English language abstract (hereinafter referred to as “Chinese-language Xiao”). '653 Prosecution History at 1274-75.

75. On November 21, 2018, the Patent Office issued a communication rejecting some of the pending claims as anticipated by, or obvious over, JP2013-019030 (Tashiharu). Other pending claims were rejected solely as being obvious over Tashiharu JP'030. *Id.* at 624-633.

76. Patent Owner responded to these anticipation and obviousness rejections over JP'030 (Tashiharu) that they were moot in view of Patent Owner's contemporaneous amendments to the claims and, with respect to a newly added claim, argued that the CaMgSi phase in the

Attorney Docket No. 2189.006

Patent 10,329,653

magnesium alloy of JP'030 is likely not galvanically-active and would not corrode in the presence of a KCL water mixture because it discloses a magnesium alloy which is usable in “transport-airplane machines” and other materials of an automobile, an airplane and a rail car, a machine part, and a robot part article and that *“a person having ordinary skill in the art would understand that the magnesium alloys . . . either improve corrosion resistance or are corrosion resistant.”* Id. at 606–07 (emphasis added).

77. The Examiner issued a Notice of Allowance on March 27, 2019, stating that claims were allowable because “the closest prior art of record,” JP2013-019030 (Tashiharu) does not teach or suggest the additive including copper, nickel, titanium, iron, or cobalt, or mixtures thereof. *Id.* at 90–91. The ‘653 Patent issued soon thereafter.

## V. CLAIM CONSTRUCTIONS

78. I have been told in the related district court litigation that Patent Owner proposed construing the various claim terms of the ‘653 Patent either using their plain and ordinary meaning to a POSITA except with respect to the following claim terms where Patent Owner has proposed (sometimes in the alternative) the following constructions (some of which were also agreed upon with the Petitioner):

<u>Claim Term</u>	<u>Definition</u>
Additive material	a material that is added
Composite	a material having two or more different elements
Controlled dissolving	dissolving a substance in a controlled manner
Forming precipitant	“forming” = “coming into existence” “precipitant” = “something having a new phase different from what existed before”

Attorney Docket No. 2189.006

Patent 10,329,653

Galvanically-active	allowing for an electrochemical reaction
in situ	in place
Intermetallic phase	Compound that has two or more metals.
melting point temperature	temperature at which liquid is formed
morphology of said galvanically-active intermetallic phases	shape of said galvanically-active intermetallic phases
Precipitation/precipitate	something having a new phase different from what existed before
Secondary metal	an additional metal that is added
Sufficient quantities in said galvanically-active intermetallic phases	"sufficient quantities" = "Enough additive material" "galvanically-active" = "allowing for an electrochemical reaction" "intermetallic phases" = "Compound that has two or more metals"
Unalloyed additive material	portion of the additive material that does not form a new compound

79. I understand that during reexamination of an unexpired patent, the USPTO's Patent and Trial Appeal Board will apply the broadest reasonable interpretation consistent with the specification standard. I also understand that the district court in the related litigation has issued a claim construction. Although I do not necessarily agree that a POSITA in April 2014 would

Attorney Docket No. 2189.006

Patent 10,329,653

construe any of the foregoing claim terms from the ‘653 Patent in the manner proposed by the Patent Owner (as set forth above), for purposes of the present declaration I adopt Patent Owner’s proposed constructions (where presented) or the broadest reasonable interpretation (where no proposal has been offered by Patent Owner) in offering my opinions below. It is my opinion that under any of these claim constructions or standards, and certainly “broadest reasonable interpretation,” the Challenged Claims are invalid for the reasons set forth below.

## **VI. OVERVIEW OF THE PRIMARY PRIOR ART**

### **A. Certified English-language translation of Chinese Patent Publication No. CN 103343271A to Xiao *et al.* (“Xiao”)**

80. I was provided with a copy of Chinese-language Patent Publication No. CN103343271A accompanied by an English language abstract (hereinafter referred to as “Chinese-language Xiao”). I noted above that Chinese-language Xiao was provided to the patent examiner during the prosecution of the ‘653 Patent.

81. I do not read, speak, or understand the Chinese language.

82. I was also provided with Xiao, a certified English translation of the Chinese-language Xiao, that I was told Petitioner commissioned (“Xiao”). I base my opinions regarding the disclosure of the Xiao reference on this certified English translation, which I exclusively reference below.

83. Xiao was filed on July 8, 2013, is titled “Light and pressure-proof fast-decomposed cast magnesium alloy,” and was published on October 9, 2013. Xiao (cover page). Because Xiao was published prior to the effective filing date of the invention of the ’653 Patent, i.e. April 18, 2014, I have been told that Xiao is prior art.

84. According to the translation, Xiao discloses a “light and pressure-proof fast-decomposed cast magnesium alloy, which can be used as a tripping ball material for a multi-stage

Attorney Docket No. 2189.006

Patent 10,329,653

sliding sleeve staged-fracturing technique, and belonging to the technical field of magnesium alloy preparation.” Xiao, ¶ 0001. Xiao explains that “[i]n recent years, the United States has made breakthroughs in the technologies of shale gas exploration and development, and the output has grown rapidly,” and that “[t]he multi-stage sliding sleeve staged fracturing technology is a new oil and gas reservoir reconstruction technology developed in recent years in the area of oil and gas well engineering technology.” *Id.* at ¶ 0002.

85. Xiao continues: “One of the key parts of this technique is the tripping ball. The tripping ball has two main functions: 1) to open the sliding sleeve to fracturing the formation rock; and 2) to separate from the fracturing fluid and pressure.” *Id.* “After the completion of all rock fracturing, it is necessary to depressurize the well tubing to facilitate the later production of the oil and gas well. In the past, the conventional method is to use the low-layer pressure to push the ball back to the well opening, or use a drilling tool to drill the sliding sleeve ball seat and the tripping ball, so as to destroy them.” *Id.*

86. Xiao identifies shortcomings of the traditional methods, i.e. the possibility that the tripping ball is stuck, delay in construction, and increase in construction costs and risks, and the need for an alternative: “the development of a tripping ball that can withstand the high pressure of fracturing construction and the high temperature at the bottom of the well, and has the feature of self-decomposition in the fluid environment of a well, can effectively reduce the construction risk and improve the construction efficiency.” *Id.*

87. Xiao sought to solve this problem by proposing a “method for preparing a light and pressure-proof fast-decomposed cast magnesium alloy by means of optimizing the alloy composition design using a self-made flux containing a rare earth element and adopting a smelting casting method.” Xiao at ¶ 0004. To do so, Xiao discloses a “magnesium alloy with a high

Attorney Docket No. 2189.006

Patent 10,329,653

aluminum content (13 to 25% by weight) and a high zinc content (2 to 10% by weight), and further adds elements of Fe, Cu, Ni and Ag which can enhance the corrosion performance of the magnesium alloy, and at the same time adds Zr, Ti element as grain refiners to improve the compressive strength of the formed material, in which the purpose of adding the high content of aluminum content is to produce a large amount of cathode phase, the  $\beta$  ( $Mg_{17}Al_{12}$ ) phase and eutectic phase on the grain boundary of the magnesium alloy, and the magnesium matrix  $\alpha$  phase functions as an anode phase, so that the matrix and the grain boundary of the magnesium alloy form a large amount of micro-batteries, which greatly accelerate the corrosion decomposition of magnesium alloy.” *Id.* at ¶ 0026.

88. Xiao explains the purpose of adding other materials to the magnesium alloy: “Elements such as Fe, Cu, Ni, Ag, etc., in the magnesium alloy can form a large number of intermetallic composite micro-particles, which can improve the corrosion performance of the magnesium alloy, thereby promoting the decomposition of the magnesium alloy.” *Id.* Thus, Xiao--unlike the prior art JP’030 (Tashiharu) reference focused on by the patent examiner--views an increased dissolution (i.e., decomposition) rate as an improvement.

89. Xiao further notes there were orders of magnitude increases in the dissolution/decomposition rate promoted by the addition of those materials (e.g. Fe, Cu, Ni, Ag, etc.) to the magnesium of: “60 to 200 times of that of the existing AZ91D magnesium alloy [at 70°C in a 3% KCl solution], and . . . 50 to 200 times [at 93°C in a 3% KCl solution] of that of the existing AZ91D magnesium alloy.” Xiao at ¶ 0026.

90. Xiao then discloses a “comparative example alloy [that] is AZ91D magnesium alloy,” *id.* at ¶¶ 0030–0032, and seven additional examples containing not only magnesium,

Attorney Docket No. 2189.006

Patent 10,329,653

aluminum, and zinc, but also Iron (Fe), Copper (Cu), Nickel (Ni), Silver (Ag), or and Titanium (Ti), *id.* at ¶¶ 0033–0060.

91. We compared the English-language abstract provided to the patent examiner with the “Abstract of the Disclosures” section of the Certified English-language translation of Xiao using the compare feature of MS Word. In my opinion, the comparison made it easier to confirm that the abstracts would have been considered practically identical from the standpoint of a POSITA in April 2014.

92. Most importantly, however, those Abstracts (Xiao) failed to disclose the truly important aspects of the subject matter of the Xiao reference. In particular, the Abstracts say nothing about galvanic-activity, corrosion, or accelerating the dissolution rate of magnesium. Rather, the Abstracts specifically state that “the alloy decomposition performance exceeds that of a traditional cast magnesium alloy.” See, Xiao. This portion of the final sentence of the Xiao Abstract likely led the patent examiner to incorrectly believe that Xiao--like much of the magnesium alloy prior art (see, e.g., ‘653 Patent, 3:19-24) including Tashiharu JP’030 considered by the patent examiner to be the closest prior art reference--disclosed, in the words of Patent Owner, “magnesium alloys [that] either improve corrosion resistance or are corrosion resistant.” (Prosecution History) at 606–07.

**B. “Development of high strength magnesium based composites using elemental nickel particulates as reinforcement,” Printed Publication by S.F. Hassan and M. Gupta (“Hassan”)**

93. Hassan, was first published in the June 2002 issue of the Journal of Materials Science, resulting in the following citation: Hassan, S.F., Gupta, M., Development of high strength magnesium based composites using elemental nickel particulates as reinforcement. *Journal of Materials Science* 37, 2467–2474 (2002). Hassan, cover page. Journal of Materials Science was

Attorney Docket No. 2189.006

Patent 10,329,653

before then and is still now considered a reputable, widely known scientific journal that has been readily accessible to POSITAs well before April 2014 (there are even issues of this Journal readily available from before 1970).

94. Hassan bears a copyright date of 2002. *Id.* Moreover, Hassan was marked “Received 14 June 2001 and accepted 12 February 2002” by its then publishing company, Kluwer Academic Publishers. Hassan, cover page and p. 2474. Volume 37, which contained Hassan, was assigned journal ISSN number (0022-2461) and a DOI number (Digital Object Identifier used for publications) “<https://doi.org/10.1023/A:1015475103720>”. *Id.* Hassan was also cited by others skilled in the art prior to April 18, 2014. (See Hai, at 18; ,Hemanth at 11.)” Because Hassan was published prior to the effective filing date of the invention of the ’653 Patent, i.e. April 18, 2014, I have been told that Hassan is prior art.

95. Hassan recognized that “[m]agnesium based materials due to their inherently low density and ensuing potential to exhibit high specific mechanical properties are actively sought for weight-critical structural application” and that “[t]he increasing hostile service conditions that the modern engineering devices have to withstand have led the materials scientist across the globe to create new materials with enhanced properties when compared to the conventional materials. One way to improve the properties of conventional metallic materials is to reinforce them judiciously keeping the end application in mind. Among the reinforced metallic materials, magnesium based composites are becoming the strong candidates for lightweight structural application due to their superior specific mechanical properties.” Hassan at 2467.

96. Hassan discloses the “synthesis of monolithic and nickel reinforced magnesium composites (Mg/Nip),” including “superheating the magnesium turnings with reinforcement

Attorney Docket No. 2189.006

Patent 10,329,653

particulates (placed in multi-layer sandwich form) to 750°C” and stirring the “molten slurry.” *Id.* at 2468.

97. Hassan describes its study as “magnesium turnings of 99.9+% purity . . . were used as the base material and elemental nickel particulates of 99.9% purity (Johnson Matthey, MA, USA) with an average size of 29±19 µm were used as reinforcement phase.” *Id.* at 2468.

98. Hassan further discloses the results for the study revealed “[s]evere reaction between magnesium melt and nickel particulates during DMD processing led to the reduction of the particulate size and formation of Mg<sub>2</sub>Ni intermetallics. The results of quantitative determination of unreacted nickel (see Table I), microstructural characterization illustrating the presence of reaction products (see Fig. 1) and XRD results (see Table II) showing the presence of Mg<sub>2</sub>Ni supports the experimental observations.” *Id.* at 2472.

**TABLE I** Results of density and porosity measurements

Material	Reinforcement in composites				Density (g/cm <sup>3</sup> )
	(wt%)	(vol%) <sup>a</sup>	(vol%) <sup>b</sup>	Size <sup>c</sup>	
Mg	—	—	—	—	1.7395 ± 0.0005
Mg/7.3Ni <sub>P</sub>	7.3	1.5	0.1	2.7 ± 0.6	1.9046 ± 0.0038
Mg/14.0Ni <sub>P</sub>	14.0	3.1	1.3	2.5 ± 0.4	2.0677 ± 0.0002
Mg/24.9Ni <sub>P</sub>	24.9	6.1	4.3	9.0 ± 4.6.	2.3834 ± 0.0092

<sup>a</sup>Computed by using the total amount of Ni retained in composite samples.

<sup>b</sup>Indicates the amount of Ni in the unreacted form present in composite samples.

<sup>c</sup>Result of cumulative image analysis conducted on fifteen representative SEM micrographs for each composite.

Attorney Docket No. 2189.006

Patent 10,329,653

**T A B L E II** Results of X-ray diffraction studies and coefficient of thermal expansion analysis

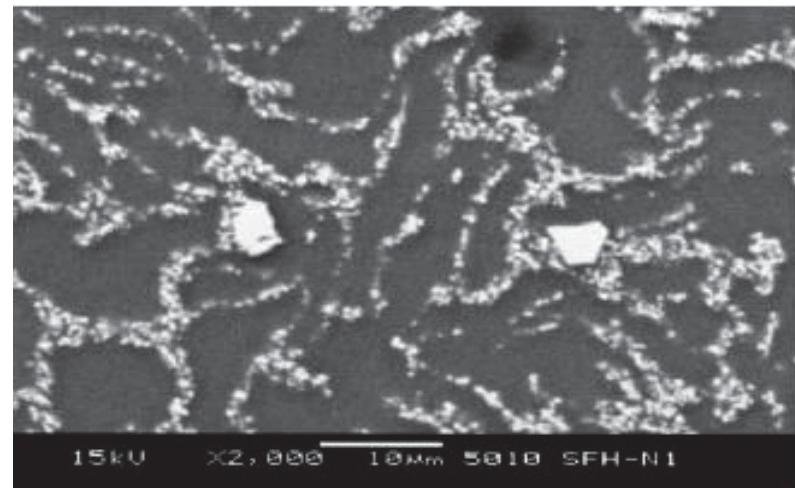
Materials	Number of matching peaks					CTE ( $\times 10^{-6}/^{\circ}\text{C}$ )
	Mg	Ni	$\text{Mg}_2\text{Ni}$	$\text{NiO}$	$\text{Ni}_2\text{O}_3$	
Mg	9 [3]	—	—	—	—	$28.60 \pm 0.07$
Mg/7.3Nip	6 [3]	—	6 [2]	—	—	$27.54 \pm 0.26$
Mg/14.0Nip	5 [3]	—	8 [2]	—	—	$26.35 \pm 0.09$
Mg/24.9Nip	6 [3]	2 [1]	16 [2]	1 [1]	1	$20.75 \pm 0.56$

[ ] indicates the number of main peaks matched.

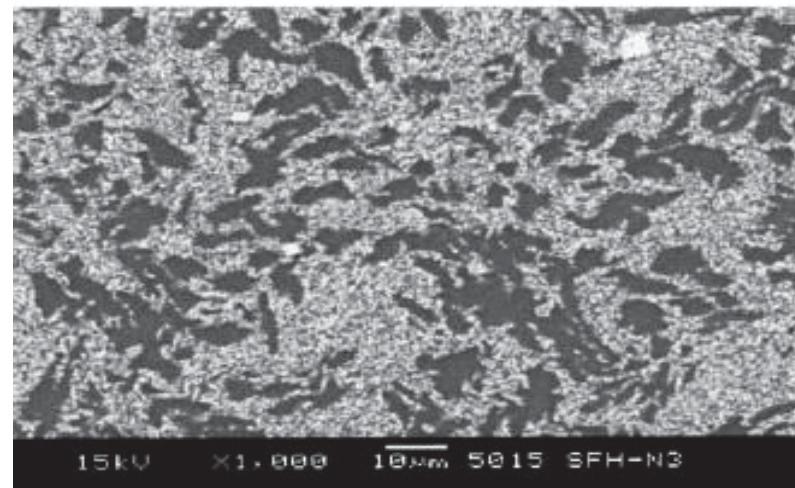
99. Figure 1 of Hassan demonstrates “representative SEM micrographs showing distribution of nickel and its reaction product in: (a) Mg/7.3Nip, (b) Mg/14.0Nip and (c) Mg/24.9Nip matrices.”

Attorney Docket No. 2189.006

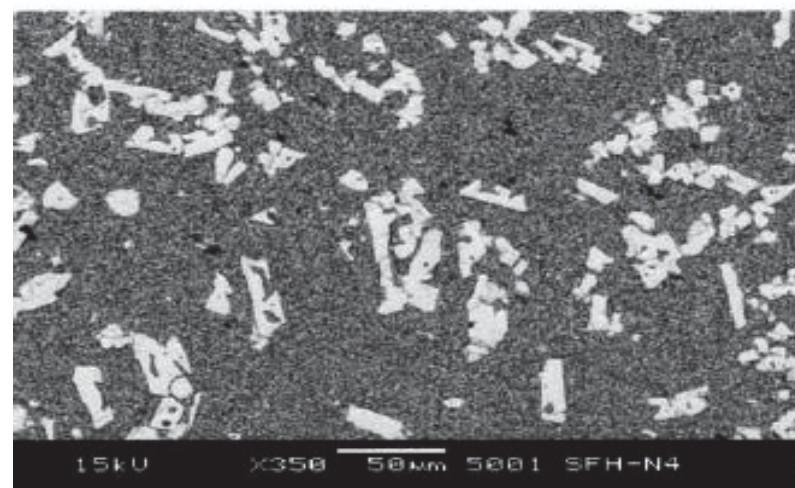
Patent 10,329,653



(a)



(b)



(c)

Attorney Docket No. 2189.006

Patent 10,329,653

100. Hassan was not considered by the patent examiner during the prosecution of the ‘653 Patent.

## **VII. SPECIFIC GROUNDS FOR PETITION**

101. Below I provide my analysis of the foregoing prior art as compared to various claims of the ‘653 Patent. For the convenience of the Board, I have color-coded specific disclosures in the prior art to corresponding limitations or requirements of the claims of the ‘653 Patent.

102. In particular I have concluded there are three bases for invalidating the Challenged Claims of the ‘653 Patent. Ground I: Claims 1,<sup>13</sup> 4, 5, 11, 13, **29, 37, 41, 43, 45, 49,** 69, 70, and **73** are anticipated by Xiao. Ground II: Claims 8, **25, 33, 55,** and **74** are anticipated, or at least made obvious, by Xiao. And Ground III: Claims 7, **12,** 13, and 71 are obvious over Xiao in view of Hassan.

### **A. Ground I: Claims 1, 4, 5, 11, 13, 29, 37, 41, 43, 45, 49, 69, 70, 71, and 73 are Anticipated by Xiao**

103. It is my opinion that Xiao anticipates Claims 1, 4, 5, 11, **12,** 13, **29, 33, 37, 41, 43, 45, 49,** 69, 70, 71, and **73** of the ‘653 Patent.

#### **1. Independent Claim 1**

##### **a. “A magnesium composite”**

104. Xiao discloses a “**magnesium alloy**...and further adds elements of Fe, Cu, Ni, and Ag which can enhance the corrosion performance of the magnesium alloy...” Xiao at ¶ 0026, ll. 1–9.

---

<sup>13</sup> I have tried to indicate which Challenged Claims are independent throughout my declaration by indicating them in bold font.

Attorney Docket No. 2189.006

Patent 10,329,653

105. Thus, Xiao's disclosure of a combination of a magnesium alloy and elements of iron, copper, nickel, and silver thus meets the construction of "a magnesium composite."

- b. "[magnesium composite] that includes **in situ precipitation of galvanically-active intermetallic phases to enable controlled dissolution of said magnesium composite,**"

106. Xiao discloses that its invention adopts a magnesium alloy with high aluminum and zinc content and **further adds elements Fe, Cu, Ni and Ag which can form intermetallics and enhance the corrosion performance of the magnesium alloy.** Xiao explains the corrosion mechanism within a magnesium alloy in terms of a cathode phase, which Xiao explains includes "the  $\beta$  ( $Mg_{17}Al_{12}$ ) phase and eutectic phase on the grain boundary of the magnesium alloy," and "the magnesium matrix  $\alpha$  phase which functions as an anode phase, so **that the matrix and the grain boundary of the magnesium alloy form a large amount of micro-batteries, which [micro-batteries] greatly accelerate the corrosion decomposition of magnesium alloy.**" *Id.* at ¶ 0026.

107. Xiao further discloses that "[e]lements such as Fe, Cu, Ni, Ag, etc., in the magnesium alloy **can form a large number of intermetallic composite micro-particles**, which can improve the corrosion performance of the magnesium alloy, thereby promoting the decomposition of the magnesium alloy." Xiao at ¶ 0026, ll. 15–18. A POSITA in August 2014 would have understood this disclosure of "intermetallic composite micro-particles" to be a reference to intermetallic precipitate.

108. A POSITA reading the Xiao reference in April 2014 would have recognized that the precipitation of galvanically-active intermetallic phases ("micro-batteries") are naturally and necessarily generated by the *in situ* reaction between magnesium and any and all of the disclosed

Attorney Docket No. 2189.006

Patent 10,329,653

metal additives forming *in situ* precipitate. Thus, all of the limitations of this claim element are disclosed by Xiao.

c. “**said magnesium composite comprising a mixture of magnesium or a magnesium alloy and an additive material,**”

109. Xiao discloses “first loading **pure magnesium and pure aluminum** into a smelting furnace . . . , **next loading** pure zinc and **an [sic] intermediate alloys** of trace element components into a **resulting magnesium-aluminum alloy melt** after melting.” *Id.* at ¶ 0022.

110. Xiao discloses that these “intermediate alloys” are **Al-Fe, Al-Ni, Al-Cu, Al-Ag, Al-Zr, Al-Ti**, which are heated to dry and then added to the magnesium-aluminum alloy melt.” Xiao at ¶ 0023. These intermediate alloys meet Patent Owner’s proposed definition of “additive material,” i.e. “a material that is added.”

111. The addition of one or more of these intermediate alloys to the melted magnesium alloy necessarily results in a mixture of magnesium or magnesium alloy and the additive material. Thus, Xiao discloses all of the limitations of this claim element.

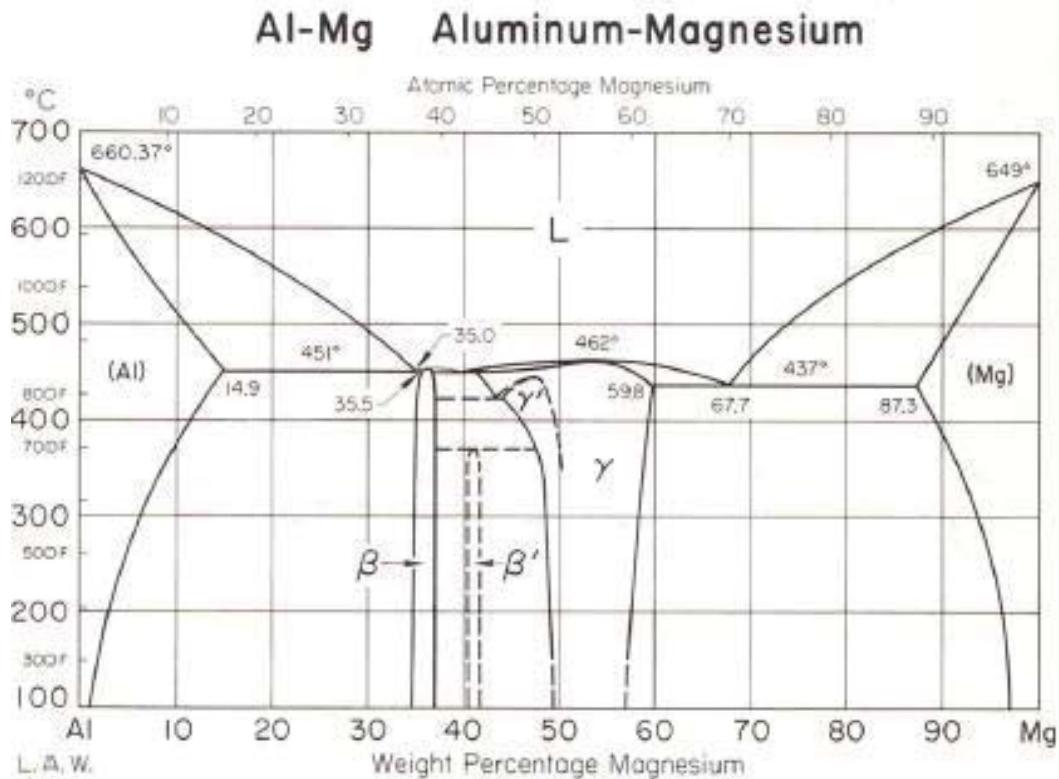
d. “**said additive material having a greater melting point temperature than a solidus temperature of said magnesium, [.. . said additive material includes one or more metals selected from the group consisting of copper, nickel, iron, and cobalt]**”

112. **All of the prior elements of Independent Claim 1 had referred to “magnesium composite” rather than just “magnesium,”** which the claim defined as comprising “a mixture of magnesium or magnesium alloy (and an additive material).” It is my opinion that a POSITA would have understood the reference to “magnesium” in this claim element as reference to the previous limitation of “magnesium or magnesium alloy.”

Attorney Docket No. 2189.006

Patent 10,329,653

113. Using a typical phase diagram, a POSITA in April 2014 would have readily determined that the solidus temperature of magnesium is the temperature at which a metal first begins to melt during heating and not necessarily when the metal is completely liquidus.



The POSITA would also use this phase diagram to find the solidus temperature of the magnesium-aluminum alloy disclosed in Xiao to be in a range of 650°C for pure magnesium and down to 437°C for aluminum additions up to 40wt%. The solidus temperature is 437°C for the magnesium-aluminum alloys disclosed in Xiao having in the range of 13 wt% to 25 wt% aluminum.

114. The **additive materials** disclosed in Xiao that were noted above are one or more of the **intermediate alloys: Al-Fe, Al-Ni, Al-Cu, Al-Ag, Al-Zr, Al-Ti**. Xiao at ¶ 0023. Patent Owner has taken the position that “melting point temperature” would have meant “temperature at which liquid is formed” to a POSITA in April 2014.

Attorney Docket No. 2189.006

Patent 10,329,653

115. The POSITA using typical phase diagrams for each of the additive materials (here the “intermediate alloys” disclosed in Xiao) would be able to readily determine the melting point temperature for each intermediate alloy. For example, the **Al-Fe** phase diagram shows a variable melting point temperature ranging from 1538°C (pure iron) down to 655°C for a eutectic temperature between about 63 wt% aluminum to near 99 wt% aluminum. The **Al-Ni** phase diagram shows a variable melting point temperature that ranges from 1640°C down to 640°C depending upon the alloy content. The **Al-Cu** phase diagram shows a variable melting point temperature ranging from 548°C up to 1084°C depending upon the alloy mixture. A POSITA in April 2014 would have readily determined the specific melting temperature of a specific alloy mixture based on these phase diagrams.

116. Xiao discloses a variety of **Al-Fe**, **Al-Ni**, **Al-Cu**, **Al-Ag**, **Al-Zr**, **Al-Ti alloy mixtures** with a variety of constituencies. For instance, Xiao specifically teaches in Example 7 that:

The composition of the alloy and the respective percentages by weight are as follows: 25% Al-10% Zn-1% Fe-0.5% Ni-0.1% Cu-0.5% Ti-0.05% Zr, and the remainder is Mg.

Xiao at ¶ 0059. Xiao teaches that the magnesium alloy of Example 7, as a whole, ends up with the maximum amount of aluminum (i.e. 25 wt%) taught as being within the invention of Xiao (see, Xiao ¶ 0026). Xiao specifically discloses--with respect to Example 7--that portions of that aluminum content are added into a magnesium-aluminum melt as an **Al-Fe intermediate alloy**, an **Al-Ni intermediate alloy**, an **Al-Cu intermediate alloy**, an **Al-Ag intermediate alloy**, an **Al-Zr intermediate alloy**, and an **Al-Ti intermediate alloy**. Xiao at ¶ 0060. Thus, it is necessary to prepare mass balance calculations to determine the relative concentrations of the pure magnesium

Attorney Docket No. 2189.006

Patent 10,329,653

and pure aluminum initially loaded into the smelting furnace in order to determine its solidus temperature.

117. First, a POSITA in April 2014, would have converted the weight percentages to grams to perform mass balances. As a result, a POSITA would have understood in April 2014 that Example 7 of Xiao includes 62.85 grams of magnesium (i.e., 100 grams - 37.15 grams (i.e., 25 grams (Al) -10 grams (Zn)- 1 gram (Fe) - 0.5 grams (Ni) - 0.1 gram (Cu) - 0.5 grams (Ti) - 0.05 grams (Zr))).

118. The following intermediate alloy compositions were chosen for the initial mass balance calculations: Al-Fe (30 wt% Fe), Al-Ni (45 wt% Ni), Al-Cu (40 wt% Cu), Al-Ti (10 wt% Ti), Al-Zr (20 wt% Zr) alloy mixtures.

119. As would a POSITA in April 2014, (using the weight percentages-to-grams conversion noted above) I performed the initial mass balances.

120. Thus, using the initially selected Al-30Fe, Al-45Ni, Al-40Cu, Al-10Ti, and Al-20Zr alloys would have resulted in 7.79 grams of Aluminum being added to the overall alloy as components of the intermediate alloys (as opposed to the aluminum introduced into the ultimate alloy of Example 7 in the initial melt). Since Example 7 of Xiao calls for a total of 25 grams (i.e. 25 wt%) of Aluminum, 17.21 grams of Aluminum would have had to have been initially loaded into the smelting furnace in Example 7 along with the 62.85 grams of magnesium (see calculation above). Thus, the initial melt of Example 7 (using the intermediate alloys selected) would have had a composition of 78.5 wt% Mg and 21.5 wt% Al.

121. Next, a POSITA would have consulted the Mg-Al phase diagram to determine that the solidus temperature of the initial magnesium alloy (i.e. 78.5 wt% Mg and 21.5 wt% Al) of Example 7 of Xiao to be 437 °C.

Attorney Docket No. 2189.006

Patent 10,329,653

122. A POSITA would also consult the relevant phase diagram to determine the melting point temperature of the various additive materials disclosed in Xiao. An aluminum-iron alloy with 30 wt% iron would have a melting point temperature of 655 °C, an aluminum-nickel alloy with 45 wt% Ni would have a melting point temperature of 854 °C, an aluminum-copper alloy with 40 wt% Cu would have a melting point temperature of 548°C, an aluminum-titanium alloy with 10 wt% Ti would have a melting point temperature of 665°C, and an aluminum-zirconium alloy with 20 wt% Zr would have a melting point temperature of 661°C. Thus, each of these additive materials from Example 7 of Xiao (with the constituencies selected above) would have had a greater melting point temperature than the solidus temperature of magnesium or the magnesium-aluminum alloy (i.e. 437°C) corresponding to 21.5 wt% aluminum which is the calculated amount of pure aluminum in pure magnesium initially loaded into the smelting furnace in Example 7. A POSITA in April 2014 would have also recognized that other compositions would have fallen within the teachings of Example 7 of Xiao as well as the other teachings of Xiao and resulted in **Al-Fe, Al-Ni, and Al-Cu intermediate alloys** having greater melting point temperatures than the solidus temperature of initial magnesium alloy loaded in the various Examples of Xiao. Thus, Xiao discloses all of the limitations of this claim element.

- e. “**said additive material constituting about 0.05 wt %-45 wt % of said mixture, [... said additive material includes one or more metals selected from the group consisting of copper, nickel, iron, and cobalt]**”

123. Claim 1 of Xiao discloses: a “magnesium alloy, comprising the components at the weight percentages as follows: **Al: 13 to 25%, Zn: 2 to 15%**, the remainder is Mg, and a sum of the weight percentages of the components is 100%.” Xiao at Claim 1. And Claim 2 discloses: a “magnesium alloy, according to Claim 1, further comprising the trace elements at the weight

Attorney Docket No. 2189.006

Patent 10,329,653

percentages as follows: **Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%**, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%; and a sum of the weight percentages of the components is 100%.” *Id.* at Claim 2.

124. These metallic elements are found in the additive materials generally disclosed by Xiao (*i.e.*, **intermediate alloys: Al-Fe, Al-Ni, Al-Cu, Al-Ti**) (Xiao at ¶ 0023) and particularly described in association with Example 7 of Xiao (*Id.* at ¶¶ 0057 - 0060) for which I have already calculated (and set forth above) the specific weight percentage (ranges) of each additive material (which has been defined by the Patent Owner as “a material that is added” (see Paragraph 78 of my Declaration)) set forth in Example 7 of Xiao (*i.e.* Al-Fe, Al-Ni, Al-Ag, Al-Cu, Al-Zr, Al-Ti and Zinc).

125. In particular, the additive materials aluminum-iron with 30 wt% iron, aluminum-nickel with 45 wt% nickel, and aluminum-copper with 40 wt% copper, which each have greater melting point temperatures than the solidus temperature of magnesium or the magnesium aluminum alloy disclosed in Xiao, also fall well within the range of about 0.05 wt% to 45 wt% of the mixture. For example, the aluminum-iron additive constitutes 3.3 wt% of the mixture (*i.e.*, 1%/0.30); the aluminum-nickel additive constitutes 1.1 wt% of the mixture (*i.e.*, 0.5%/0.45); and the aluminum-copper additive constitutes 0.25 wt% of the mixture (*i.e.*, 0.1%/0.40). A POSITA in April 2014 would have also recognized that other intermediate alloy compositions would have fallen within the teachings of Example 7 of Xiao as well as the other teachings of Xiao and resulted in **Al-Fe, Al-Ni, and Al-Cu intermediate alloys** constituting about 0.05 wt% to 45 wt% of the mixture. Accordingly, this element of the claim is disclosed by Xiao.

f.       **“said additive material forming precipitant in said magnesium composite,”**

126. Xiao discloses that constituents of the additive materials disclosed in Xiao (and particularly disclosed in Example 7, thereof) form precipitant: “**Elements such as Fe, Cu, Ni, Ag,**

Attorney Docket No. 2189.006

Patent 10,329,653

**etc.**, in the magnesium alloy **can form a large number of intermetallic composite micro-particles**, which can improve the corrosion performance of the magnesium alloy, thereby promoting the decomposition of the magnesium alloy.” Xiao at ¶ 0026; see also ¶¶ 0057-0060 (Example 7).

127. As established in the section above, the intermediate alloys Al-Fe and Al-Ni disclosed in Xiao have greater melting point temperatures than the solidus temperature of magnesium or the magnesium aluminum alloy disclosed in Xiao. Those alloys would necessarily contribute FeAl<sub>3</sub>, Al<sub>3</sub>Ni, Al<sub>3</sub>Ni<sub>2</sub>, and/or Mg<sub>2</sub>Ni to the magnesium composite, which Xiao teaches would lead to the formation of micro-particles, also known as precipitant. Thus, this element of the claim is disclosed by Xiao.

g. **“said additive material includes one or more metals selected from the group consisting of copper, nickel, iron, and cobalt,”**

128. Claim 2 of Xiao discloses a “magnesium alloy according to Claim 1, further comprising the trace elements at the weight percentages as follows: **Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%**, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%; and a sum of the weight percentages of the components is 100%.” Thus, the invention of Xiao generally teaches additive materials containing **iron, copper, and nickel**. Of the additive materials taught in Example 7 of Xiao (i.e. Al-Fe, Al-Ni, Al-Ag, Al-Cu, Al-Zr, Al-Ti and Zinc), **Al-Fe, Al-Ni, and Al-Cu** contain **copper, nickel and iron**. This claim element requires the additive material include even only one of these metals. Claim 2 and Example 7 both teach three. Accordingly, Xiao discloses this element of the claim.

Attorney Docket No. 2189.006

Patent 10,329,653

- h. “said magnesium composite has a dissolution rate of at least 5 mg/cm<sup>2</sup>/hr. in 3 wt % KCl water mixture at 90°C.”**

129. A POSITA in April 2014 would have understood the English translation of Xiao to disclose a table of dissolution rates (“decomposition rates”) because the units of measurement in that table are units regularly used by POSITAs to express measurements of dissolution. In particular, Xiao provides dissolution rates for its various examples, as follows:

	Room temperature tensile strength $\sigma_b$ (MPa)	Decomposition rate at 70 °C in 3% KCl solution (g.cm <sup>-2</sup> .h <sup>-1</sup> )	Decomposition rate at 93 °C in 3% KCl solution (g.cm <sup>-2</sup> .h <sup>-1</sup> )
Comparative example 1	232	0.00026	0.0005
Example 1	360	0.035	0.074
Example 2	385	0.015	0.045
Example 3	410	0.013	0.036
Example 4	375	0.034	0.058
Example 5	392	0.025	0.048
Example 6	365	0.021	0.063
Example 7	387	0.036	0.057

Xiao at ¶ 0064.

130. Xiao provides dissolution rates in g/cm<sup>2</sup>/hr, while the claims of the '653 patent express the claimed dissolution rate in mg/cm<sup>2</sup>/hr. A POSITA in April 2014 would have known that converting from g/cm<sup>2</sup>/hr to mg/cm<sup>2</sup>/hr, merely requires multiplication of the numbers disclosed in this table from Xiao by 1,000.

131. Thus, the dissolution rates disclosed in Xiao convert to:

Attorney Docket No. 2189.006

Patent 10,329,653

	Decomposition rate at 70°C in 3% KCl solution mg/cm <sup>2</sup> /hr	Decomposition rate at 93°C in 3% KCl solution mg/cm <sup>2</sup> /hr
Comparative example 1	0.26	0.5
Example 1	35	74
Example 2	15	45
Example 3	13	36
Example 4	34	58
Example 5	25	48
Example 6	21	63
Example 7	36	57

132. Each of the seven examples (excluding the comparative [prior art] example) provided in Xiao therefore discloses a dissolution rate of “at least 5 mg/cm<sup>2</sup>/hr in 3 wt.% KCl water” at both 70°C or 93°C. Given that the disclosed dissolution rates of Xiao both above and below 90°C are well above the claimed floor of “at least 5 mg/cm<sup>2</sup>/hr” (i.e. at least three times overall and for Example 7 at least seven times) it is my opinion that a POSITA in April 2014 would have recognized the dissolution rate of the examples in Xiao at 93°C to also be necessarily well above the claimed floor of “at least of 5 mg/cm<sup>2</sup>/hr.” Consequently, this element of Claim 1 is also disclosed by Xiao.

133. Accordingly, as set forth in the immediately foregoing paragraphs, it is my opinion that Xiao would have fully disclosed each and every element of Claim 1 of the ‘653 Patent to a POSITA in or before April 2014 in accordance with Patent Owner’s definitions (set forth in Paragraph 78 of my Declaration).

Attorney Docket No. 2189.006

Patent 10,329,653

## 2. Dependent Claim 4

- a. “The magnesium composite as defined in claim 1, wherein said additive material includes nickel,”

134. As set forth above, it is my opinion that Xiao would have disclosed all elements of Claim 1 to a POSITA in April 2014.

135. The overall teaching of Xiao notes the use of nickel as an additive material. See, e.g., Xiao at ¶ 0026. As I discussed above, Example 7 of Xiao particularly discloses intermediate alloys Al-Fe, Al-Ni, Al-Ag, Al-Cu, Al-Zr, Al-Ti and Zinc are used as additive materials. A POSITA in April 2014 would have understood that the intermediate alloy **Al-Ni contains nickel**. Accordingly, Example 7 of Xiao discloses this element, in accordance with Patent Owner’s definition of “additive material.”

- b. “said nickel constitutes about 0.05-35 wt % of said magnesium composite,”

136. Claim 2 of Xiao discloses “**Ni: 0.05 to 5%** . . . and a sum of the weight percentages of the components is 100%.” In particular, Example 7 of Xiao (which I analyzed and discussed above) discloses **0.5 wt% nickel**. Thus, the range of nickel disclosed by Xiao falls within the range claimed in Claim 4 of the ‘653 Patent.

- c. “said nickel forms galvanically-active in situ precipitate in said magnesium composite.”

137. Xiao discloses that “[e]lements such as Fe, Cu, **Ni**, Ag, etc., in the magnesium alloy can form a large number of **intermetallic composite micro-particles**, which can improve the corrosion performance of the magnesium alloy, thereby promoting the decomposition of the magnesium alloy.” Xiao at ¶ 0026.

138. A POSITA in April 2014 would have understood that a precipitate would necessarily be formed in place and that the “composite microparticles” disclosed by Xiao are the

Attorney Docket No. 2189.006

Patent 10,329,653

same as the claimed “precipitate” (i.e. “something having a new phase different from what existed before” under Patent Owner’s proposed definition). Xiao would have further disclosed to the POSITA in April 2014 that the corrosion rate of its novel magnesium alloys is accelerated by the formation of micro-batteries between the boundary of the matrix and the grains (i.e. intermetallic composites) of the magnesium alloy. *Id.* A POSITA in April 2014 would have understood this reference to micro-batteries to be a reference to the galvanic activity of *in situ* precipitates. Consequently, all of the limitations of Dependent Claim 4 are taught by Xiao.

### **3. Dependent Claim 5**

- a. “The magnesium composite as defined in claim 1, wherein said additive material includes copper,”**

139. As set forth above, it is my opinion that Xiao would have disclosed all elements of Claim 1 to a POSITA in April 2014.

140. The overall teaching of Xiao notes the use of copper as an additive material. See, e.g., Xiao at ¶ 0026. As I discussed above, Example 7 of Xiao particularly discloses intermediate alloys Al-Fe, Al-Ni, Al-Ag, Al-Cu, Al-Zr, Al-Ti and Zinc are used as additive materials. A POSITA in April 2014 would have understood that the intermediate alloy **Al-Cu contains copper**. Accordingly, Example 7 of Xiao discloses this element, in accordance with Patent Owner’s definition of “additive material.”

- b. “said copper constitutes about 0.05-35 wt % of said magnesium composite,”**

141. Claim 2 of Xiao discloses “**Cu: 0.05 to 5%**, . . . and a sum of the weight percentages of the components is 100%.” In particular, Example 7 of Xiao (which I analyzed and discussed above) disclosed **0.1% copper**. Thus the range of copper disclosed by Xiao falls within the range claimed in Claim 5 of the ‘653 Patent.

Attorney Docket No. 2189.006

Patent 10,329,653

c.     “**said copper forms galvanically-active in situ precipitate in said magnesium composite.**”

142. Xiao discloses that “[e]lements such as Fe, Cu, Ni, Ag, etc., in the magnesium alloy can form a large number of **intermetallic composite micro-particles**, which can improve the corrosion performance of the magnesium alloy, thereby promoting the decomposition of the magnesium alloy.” *Id.* at ¶ 0026.

143. A POSITA in April 2014 would have understood that a precipitate would necessarily be formed in place and that the “composite microparticles” disclosed by Xiao are the same as the claimed precipitate (under Patent Owner’s proposed definition). Xiao would have further disclosed to the POSITA in April 2014 that the corrosion rate of its novel magnesium alloys is accelerated by the formation of micro-batteries between the boundary of the matrix and the grains (i.e. intermetallic composites) of the magnesium alloy. *Id.* A POSITA in April 2014 would have understood this reference to micro-batteries to be a reference to the galvanic activity of the *in situ* precipitate. Consequently, all of the limitations of Dependent Claim 5 are taught by Xiao.

#### 4. Dependent Claim 11

a.     “**The magnesium composite as defined in claim 1, wherein said additive material is a metal or metal alloy.**”

144. As set forth above, it is my opinion that Xiao would have disclosed all elements of Claim 1 to a POSITA in April 2014.

145. The overall teaching of Xiao notes the use of a variety of metals as additive materials. See, e.g., Xiao at ¶ 0026. As I discussed above, Example 7 of Xiao particularly discloses intermediate alloys Al-Fe, Al-Ni, Al-Ag, Al-Cu, Al-Zr, Al-Ti and Zinc are used as additive materials. A POSITA in April 2014 would have understood that all of these intermediate alloys contain metal (**i.e., aluminum, iron, nickel, silver, copper, zirconium, and titanium**) and that

Attorney Docket No. 2189.006

Patent 10,329,653

**zinc** is a metal. Accordingly, Example 7 of Xiao discloses this element, in accordance with Patent Owner's definition of "additive material" and, thus, all of the limitations of Dependent Claim 11 are taught by Xiao.

## 5. Independent Claim 29

### a. "A dissolvable magnesium alloy composite"

146. Xiao discloses "a **magnesium alloy**...and further adds elements of **Fe, Cu, Ni, and Ag** which can **enhance the corrosion performance** of the magnesium alloy..." Xiao at ¶ 0026, ll. 1–9.

147. Thus, Xiao's disclosure of a combination of a magnesium alloy and elements of iron, copper, nickel, and silver thus meets the construction of "a dissolvable magnesium composite."

### b. "for use in a ball or other tool component in a well drilling or completion operation,"

148. Xiao discloses that the cast magnesium alloy produced "**can be used as a tripping ball material for a multi-stage sliding sleeve staged-fracturing technique.**" *Id.* at ¶ 0001. A POSITA would have understood that a tripping ball was a type of tool component in an oil well drilling operation. Thus, Xiao teaches this limitation of the claim.

### c. "said dissolvable magnesium alloy composite comprising 60-95 wt % magnesium;"

149. Claim 1 of Xiao discloses a "**magnesium alloy**, comprising the components at the weight percentages as follows: **Al: 13 to 25%, Zn: 2 to 15%, the remainder is Mg, and a sum of the weight percentages of the components is 100%.**" As such, the total range of the non-magnesium elements disclosed in Claim 1 of Xiao is 15% (*i.e.*, 13 + 2) to 40% (*i.e.*, 25 + 15), leaving **85 to 60 wt%** for the **magnesium**. The Examples of Xiao disclose a range of magnesium

Attorney Docket No. 2189.006

Patent 10,329,653

**from 54.5 wt% - 79.2 wt%.** Thus, Xiao discloses a range of magnesium of 54.5 wt% to 85 wt %, which overlaps and covers most of the claimed range. Accordingly, Xiao teaches this limitation of the claim.

d.     **[said dissolvable magnesium alloy composite comprising] 0.01-1 wt % zirconium;**"

150. Claim 2 of Xiao discloses a "magnesium alloy, comprising the components at the weight percentages as follows: Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%, **Zr: 0.05 to 0.5%**, Ti: 0.05 to 0.5%; and a sum of the weight percentages of the components is 100%." Example 7 of Xiao teaches 0.05 wt% Zirconium in a magnesium alloy. (Examples 1-6 also teach the use of Zirconium in a magnesium alloy in amounts ranging from 0.1 wt% to 0.5 wt%).) Thus, Xiao teaches zirconium within most of the claimed range. Accordingly, Xiao teaches this limitation of the claim.

e.     **"and about 0.05-45 wt % of a secondary metal [... said secondary metal including one or more metals selected from the group consisting of copper, nickel, cobalt, titanium and iron]"**

151. Claim 1 of Xiao discloses a "magnesium alloy, comprising the components at the weight percentages as follows: Al: 13 to 25%, Zn: 2 to 15%, the remainder is Mg, and a sum of the weight percentages of the components is 100%." Xiao at Claim 1. And Claim 2 of Xiao discloses that the "magnesium alloy according to claim 1, further comprising trace elements at the weight percentages as follows: **Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%**, Zr: 0.05 to 0.5%, **Ti: 0.05 to 0.5%**; and a sum of the weight percentages of the components is 100%." *Id.* at Claim 2.

152. These metallic elements are found in the additive materials generally disclosed by Xiao (*i.e.*, **intermediate alloys: Al-Fe, Al-Ni, Al-Cu, Al-Ti**) (Xiao at ¶ 0023) and particularly described in association with Example 7 of Xiao (*Id.* at ¶¶ 0057 - 0060) for which I have already

Attorney Docket No. 2189.006

Patent 10,329,653

calculated (and set forth above) the specific weight percentage (ranges) of each additive material (which has been defined by the Patent Owner as “a material that is added” (see Paragraph 78 of my Declaration)) set forth in Example 7 of Xiao (i.e. Al-Fe, Al-Ni, Al-Ag, Al-Cu, Al-Zr, Al-Ti and Zinc).

153. In particular, the additive materials aluminum-iron with 30 wt% iron, aluminum-nickel with 45 wt% nickel, and aluminum-copper with 40 wt% copper, which each have greater melting point temperatures than the solidus temperature of magnesium or the magnesium aluminum alloy disclosed in Xiao, also fall well within the range of about 0.05 wt% to 45 wt% of the mixture. For example, the **aluminum-iron** additive constitutes 3.3 wt% of the mixture (*i.e.*, 1%/0.30); the **aluminum-nickel** additive constitutes 1.1 wt% of the mixture (*i.e.*, 0.5%/0.45); and the **aluminum-copper** additive constitutes 0.25 wt% of the mixture (*i.e.*, 0.1%/0.40). A POSITA in April 2014 would have also recognized that other intermediate alloy compositions would have fallen within the teachings of Example 7 of Xiao as well as the other teachings of Xiao and resulted in **Al-Fe, Al-Ni, and Al-Cu, and Al-Ti intermediate alloys** constituting about 0.05 wt% to 45 wt% of the mixture.

154. Thus, Xiao discloses the inclusion of secondary metals in a magnesium composite within weight percentage ranges within the claimed range. Moreover, as this claim internally defines “a secondary metal” as “including one or more metals,” Xiao also discloses a range of secondary metal running **from 0.05 wt %** (*i.e.*, the low end of the **Copper, Nickel, and Iron, and Titanium** ranges) **to 15.5 wt %** (*i.e.*, the accumulation of the high end of the **Copper, Nickel, Iron, and Titanium** ranges (5 wt % + 5 wt % +5 wt%+ 0.5 wt%)). A POSITA in April 2014 would have also recognized that the accumulated weight percentages of all of the secondary metals in Example 7 of Xiao (using the alloy compositions discussed above) would have been 9.65 wt%

Attorney Docket No. 2189.006

Patent 10,329,653

(i.e., 3.3 wt% + 1.1 wt% + 0.25 wt% + 5.0 wt%), also within the claimed range. Thus, Xiao teaches secondary metals having 0.05 to 15.5 wt%. Accordingly, Xiao teaches this limitation of the claim.

- f. “[a secondary metal] to form a galvanically-active intermetallic particle that promotes corrosion of said dissolvable magnesium alloy composite,”

155. Xiao discloses that its invention it adopts a magnesium alloy with high aluminum and zinc content and further adds elements Fe, Cu, Ni and Ag which can form intermetallics and enhance the corrosion performance of the magnesium alloy. Xiao explains the corrosion mechanism within a magnesium alloy in terms of a cathode phase, which Xiao explains includes “the  $\beta$  ( $Mg_{17}Al_{12}$ ) phase and eutectic phase on the grain boundary of the magnesium alloy,” and “the magnesium matrix  $\alpha$  phase which functions as an anode phase, so that the matrix and the grain boundary of the magnesium alloy form a large amount of micro-batteries, which [micro-batteries] greatly accelerate the corrosion decomposition of magnesium alloy.” *Id.* at ¶ 0026.

156. Xiao discloses that “[e]lements such as Fe, Cu, Ni, Ag, etc., in the magnesium alloy can form a large number of intermetallic composite micro-particles, which can improve the corrosion performance of the magnesium alloy, thereby promoting the decomposition of the magnesium alloy.” *Id.* at ¶ 0026. Thus, all of the limitations of this claim element are disclosed by Xiao.

- g. “said secondary metal including one or more metals selected from the group consisting of copper, nickel, cobalt, titanium and iron,”

157. Claim 2 of Xiao discloses a “magnesium alloy according to Claim 1, further comprising the trace elements at the weight percentages as follows: **Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%**, Zr: 0.05 to 0.5%, **Ti: 0.05 to 0.5%**; and a sum of the weight percentages of

Attorney Docket No. 2189.006

Patent 10,329,653

	Room temperature tensile strength $\sigma_b$ (MPa)	Decomposition rate at 70 °C in 3% KCl solution (g.cm <sup>-2</sup> .h <sup>-1</sup> )	Decomposition rate at 93 °C in 3% KCl solution (g.cm <sup>-2</sup> .h <sup>-1</sup> )
Comparative example 1	232	0.00026	0.0005
Example 1	360	0.035	0.074
Example 2	385	0.015	0.045
Example 3	410	0.013	0.036
Example 4	375	0.034	0.058
Example 5	392	0.025	0.048
Example 6	365	0.021	0.063
Example 7	387	0.036	0.057

the components is 100%.” Of the additive materials taught in Example 7 of Xiao (i.e. Al-Fe, Al-Ni, Al-Ag, Al-Cu, Al-Zr, Al-Ti and Zinc), **Al-Fe, Al-Ni, Al-Cu, and Al-Ti** contain **copper, nickel, titanium and iron**. This claim element only requires the secondary metal include one of these metals. Thus, Xiao teaches four candidates to serve as the secondary metal of this claim: Copper (Cu), Nickel (Ni), Titanium (Ti), and Iron (Fe).

**h. “said magnesium alloy composite has a dissolution rate of at least 5 mg/cm<sup>2</sup>/hr. in 3 wt % KCl water mixture at 90°C.”**

158. A POSITA in April 2014 would have understood the English translation of Xiao to disclose a table of dissolution rates (“decomposition rates”) because the units of measurement are the units used by POSITAs to measure dissolution rates. In particular, Xiao provides dissolution rates for its various examples, as follows:

Attorney Docket No. 2189.006

Patent 10,329,653

Xiao at ¶ 0064.

159. Xiao provides dissolution rates in g/cm<sup>2</sup>/hr, while the claims of the '653 Patent express dissolution rates in mg/cm<sup>2</sup>/hr. A POSITA in April 2014 would have known that converting from g/cm<sup>2</sup>/hr to mg/cm<sup>2</sup>/hr, merely requires multiplication of the numbers disclosed in this table in Xiao by 1,000.

160. Thus, the dissolution rates disclosed in Xiao convert to:

	Decomposition rate at 70°C in 3% KCl solution mg/cm <sup>2</sup> /hr	Decomposition rate at 93°C in 3% KCl solution mg/cm <sup>2</sup> /hr
Comparative example 1	0.26	0.5
Example 1	35	74
Example 2	15	45
Example 3	13	36
Example 4	34	58
Example 5	25	48
Example 6	21	63
Example 7	36	57

161. Each of the seven examples (excluding the comparative [prior art] example) provided in Xiao therefore discloses a dissolution rate of “at least 5 mg/cm<sup>2</sup>/hr in 3 wt.% KCl water” at both 70°C or 93°C. Given that the dissolution rate disclosed in Xiao both above and below 90°C are both well above the claimed floor of “at least 5 mg/cm<sup>2</sup>/hr,” (i.e. at least three

Attorney Docket No. 2189.006

Patent 10,329,653

times overall and for Example 7 at least seven times) it is my opinion that a POSITA in April 2014 would have recognized the dissolution rate of the examples in Xiao at 93°C to also be necessarily well above the claimed floor “at least of 5 mg/cm<sup>2</sup>/hr.” Consequently, Xiao discloses this element of the claim.

162. Accordingly, as set forth in the immediately foregoing paragraphs, it is my opinion that Xiao would have fully disclosed each and every element of Claim 29 of the ‘653 Patent to a POSITA in or before April 2014 in accordance with Patent Owner’s definitions (set forth in Paragraph 78 of my Declaration).

## **6. Independent Claim 37**

### **a. “A dissolvable magnesium alloy composite”**

163. Xiao discloses a “**magnesium alloy**...and further adds elements of **Fe, Cu, Ni, and Ag** which can **enhance the corrosion performance** of the magnesium alloy...” Xiao at ¶ 0026, ll. 1–9.

164. Thus, Xiao’s disclosure of a combination of a magnesium alloy and elements of iron, copper, nickel, and silver thus meets the construction of “a dissolvable magnesium composite.”

### **b. “for use in a ball or other tool component in a well drilling or completion operation,”**

165. Xiao discloses that the cast magnesium alloy “**can be used as a tripping ball material for a multi-stage sliding sleeve staged-fracturing technique.**” *Id.* at ¶ 0001. A POSITA would have understood that a tripping ball was a type of tool component in an oil well drilling operation. Thus, Xiao teaches this limitation of the claim.

Attorney Docket No. 2189.006

Patent 10,329,653

- c.     “**said dissolvable magnesium alloy composite comprising 60-95 wt % magnesium;**”

166. Claim 1 of Xiao discloses a “**magnesium alloy**, comprising the components at the weight percentages as follows: **Al: 13 to 25%, Zn: 2 to 15%,** the **remainder is Mg, and a sum of the weight percentages of the components is 100%.**” As such, the total range of the non-magnesium elements disclosed in Claim 1 of Xiao is 15% (*i.e.*, 13 + 2) to 40% (*i.e.*, 25 + 15), leaving **85 to 60 wt%** for the **magnesium**. The Examples of Xiao disclose a range of magnesium **from 54.5 wt% - 79.2 wt%**. Thus, Xiao discloses a range of magnesium of 54.5 wt% to 85 wt %, which overlaps and covers most of the claimed range. Accordingly, Xiao teaches this limitation of the claim.

- d.     “[**said dissolvable magnesium alloy composite comprising . . .] 0.05-6 wt % zinc; 0.01-1 wt % zirconium;**”

167. Claim 1 of Xiao discloses “**Al: 13 to 25%, Zn: 2 to 15%,**” and Claim 2 of Xiao discloses secondary metals of “**Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%;** and a sum of the weight percentages of the components is 100%.”

168. Thus, Xiao discloses a dissolvable magnesium alloy composite including weight ranges of zinc (Zn) and zirconium (Zr), which overlap a portion of the ranges required by this claim element for those metallic elements. Accordingly, Xiao teaches this limitation of the claim.

- e.     “**and [said dissolvable magnesium alloy composite comprising . . .] about 0.05-45 wt % of a secondary metal [ . . . said secondary metal including one or more metals selected from the group consisting of copper, nickel, cobalt, titanium and iron]**”

169. Claim 2 of Xiao discloses a “magnesium alloy according to Claim 1, further comprising the trace elements at the weight percentages as follows: **Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%;** and a sum of the weight percentages of the components is 100%.” Thus, Xiao teaches four candidates to serve as the secondary metal

Attorney Docket No. 2189.006

Patent 10,329,653

within the claimed weight percentage [and specific metal] limitations of this claim: **Copper (Cu), Nickel (Ni), Titanium (Ti), and Iron (Fe)**. Moreover, as this claim internally defines “a secondary metal” as “including one or more metals,” Xiao also discloses a range of secondary metal running **from 0.05 wt %** (*i.e.*, the low end of either of the Copper, Nickel, or Titanium ranges) **to 15.5 wt %** (*i.e.*, the accumulation of the high end of the Copper, Nickel, Titanium, and Iron ranges (5 wt % + 5 wt % + 0.5 wt% + 5 wt%)). Hence, Xiao discloses the inclusion of secondary metal in a magnesium composite (including one or more metals (*i.e.*, copper, nickel, titanium, and iron)) in amounts that overlap at least a portion of the range required by this claim element. Accordingly, Xiao teaches this limitation of the claim.

f. **“[a secondary metal] to form a galvanically-active intermetallic particle that promotes corrosion of said dissolvable magnesium alloy composite,”**

170. Xiao discloses that its invention it adopts a magnesium alloy with high aluminum and zinc content and **further adds elements Fe, Cu, Ni and Ag which can form intermetallics and enhance the corrosion performance of the magnesium alloy**. Xiao explains the corrosion mechanism within a magnesium alloy in terms of a cathode phase, which Xiao explains includes “the  $\beta$  ( $Mg_{17}Al_{12}$ ) phase and eutectic phase on the grain boundary of the magnesium alloy,” and “the magnesium matrix  $\alpha$  phase which functions as an anode phase, so **that the matrix and the grain boundary of the magnesium alloy form a large amount of micro-batteries, which [micro-batteries] greatly accelerate the corrosion decomposition of magnesium alloy.**” *Id.* at ¶ 0026.

171. Xiao discloses that “[e]lements such as **Fe, Cu, Ni**, Ag, etc., in the magnesium alloy **can form a large number of intermetallic composite micro-particles**, which can **improve the corrosion performance of the magnesium alloy, thereby promoting the decomposition of the**

Attorney Docket No. 2189.006

Patent 10,329,653

**magnesium alloy.”** *Id.* at ¶ 0026. These elements are among the claimed “secondary metals” disclosed by Xiao. Thus, all of the limitations of this claim element are disclosed by Xiao.

- g.     **“said secondary metal including one or more metals selected from the group consisting of copper, nickel, cobalt, titanium and iron,”**

172. Xiao discloses one or more of the “**secondary metals**,” including **copper (Cu)**, **nickel (Ni)**, **titanium (Ti)** and **iron (Fe)** as follows: “**Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%**, Zr: 0.05 to 0.5%, **Ti: 0.05 to 0.5%**.**”** Xiao at Claim 2. Of the additive materials taught in Example 7 of Xiao (i.e. Al-Fe, Al-Ni, Al-Ag, Al-Cu, Al-Zr, Al-Ti and Zinc), **Al-Fe**, **Al-Ni**, **Al-Cu**, and **Al-Ti** contain **copper, nickel, titanium and iron**. This claim element only requires the secondary metal include one of these metals. Thus, Xiao teaches four candidates to serve as the secondary metal of this claim: Copper (Cu), Nickel (Ni), Titanium (Ti), and Iron (Fe).

- h.     **“said magnesium alloy composite has a dissolution rate of at least 5 mg/cm<sup>2</sup>/hr. in 3 wt % KCl water mixture at 90°C.”**

173. A POSITA in April 2014 would have understood the English translation of Xiao to disclose a table of dissolution rates (“decomposition rates”) because the units of measurement are the units used by POSITAs to measure dissolution rates. In particular, Xiao provides dissolution rates for its various examples, as follows:

Attorney Docket No. 2189.006

Patent 10,329,653

	Room temperature tensile strength $\sigma_b$ (MPa)	Decomposition rate at 70 °C in 3% KCl solution (g.cm <sup>-2</sup> .h <sup>-1</sup> )	Decomposition rate at 93 °C in 3% KCl solution (g.cm <sup>-2</sup> .h <sup>-1</sup> )
Comparative example 1	232	0.00026	0.0005
Example 1	360	0.035	0.074
Example 2	385	0.015	0.045
Example 3	410	0.013	0.036
Example 4	375	0.034	0.058
Example 5	392	0.025	0.048
Example 6	365	0.021	0.063
Example 7	387	0.036	0.057

Xiao at ¶ 0064.

174. Xiao provides dissolution rates in g/cm<sup>2</sup>/hr, while the claims of the '653 Patent express dissolution rates in mg/cm<sup>2</sup>/hr. A POSITA in April 2014 would have known that converting from g/cm<sup>2</sup>/hr to mg/cm<sup>2</sup>/hr, merely requires multiplication of the numbers disclosed in this table in Xiao by 1,000.

175. Thus, the dissolution rates disclosed in Xiao convert to:

Attorney Docket No. 2189.006

Patent 10,329,653

	Decomposition rate at 70°C in 3% KCl solution mg/cm <sup>2</sup> /hr	Decomposition rate at 93°C in 3% KCl solution mg/cm <sup>2</sup> /hr
Comparative example 1	0.26	0.5
Example 1	35	74
Example 2	15	45
Example 3	13	36
Example 4	34	58
Example 5	25	48
Example 6	21	63
Example 7	36	57

176. Each of the seven examples (excluding the comparative [prior art] example) provided in Xiao therefore discloses a dissolution rate of “at least 5 mg/cm<sup>2</sup>/hr in 3 wt.% KCl water” at both 70°C or 93°C. Given that the disclosed dissolution rates of Xiao both above and below 90°C are well above the claimed floor of “at least 5 mg/cm<sup>2</sup>/hr” (i.e. at least three times overall and for Example 7 at least seven times) it is my opinion that a POSITA in April 2014 would have recognized the dissolution rate of the examples in Xiao at 93°C to also be necessarily well above the claimed floor of “at least of 5 mg/cm<sup>2</sup>/hr.” Consequently, this element of Claim 1 is also disclosed by Xiao.

177. Accordingly, as set forth in the immediately foregoing paragraphs, it is my opinion that Xiao would have fully disclosed each and every element of Claim 37 of the ‘653 Patent to a POSITA in or before April 2014 in accordance with Patent Owner’s definitions (set forth in Paragraph 78 of my Declaration).

Attorney Docket No. 2189.006

Patent 10,329,653

## 7. Independent Claim 41

### a. “A dissolvable magnesium alloy composite”

178. Xiao discloses “a **magnesium alloy**...and further adds elements of **Fe, Cu, Ni, and Ag** which can **enhance the corrosion performance** of the magnesium alloy...” Xiao at ¶ 0026, ll. 1–9.

179. Thus, Xiao’s disclosure of a combination of a magnesium alloy and elements of iron, copper, nickel, and silver thus meets the construction of “a dissolvable magnesium composite.”

### b. “for use in a **ball or other tool component** in a well drilling or completion operation,”

180. Xiao discloses that the cast magnesium alloy “**can be used as a tripping ball material for a multi-stage sliding sleeve staged-fracturing technique.**” *Id.* at ¶ 0001. A POSITA would have understood that a tripping ball was a type of tool component in an oil well drilling operation. Thus, Xiao teaches this limitation of the claim.

### c. “**said dissolvable magnesium alloy composite comprising over 50 wt % magnesium;**”

181. Claim 1 of Xiao discloses a “**magnesium alloy**, comprising the components at the weight percentages as follows: **Al: 13 to 25%, Zn: 2 to 15%, the remainder is Mg, and a sum of the weight percentages of the components is 100%.**” As such, the total range of the non-magnesium elements disclosed in Claim 1 of Xiao is 15% (*i.e.*, 13 + 2) to 40% (*i.e.*, 25 + 15), leaving **85 to 60 wt%** for the **magnesium**. The Examples of Xiao disclose a range of magnesium **from 54.5 wt% - 79.2 wt%**. Thus, Xiao discloses a range of magnesium of 54.5 wt% to 85 wt %, which covers most of the claimed range. Accordingly, Xiao teaches this limitation of the claim.

### d. “[**said dissolvable magnesium alloy composite comprising . . .]** one or more metals selected from the group consisting of **0.5-10**

Attorney Docket No. 2189.006

Patent 10,329,653

**wt % aluminum, 0.1-2 wt % zinc, 0.01-1 wt % zirconium, and 0.15-2 wt % manganese;”**

182. Claim 1 of Xiao discloses a “magnesium alloy, comprising the components at the weight percentages as follows: Al: 13 to 25%, **Zn: 2 to 15%**,” and Claim 2 of Xiao discloses secondary metals of “Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%, **Zr: 0.05 to 0.5%**, Ti: 0.05 to 0.5%; and a sum of the weight percentages of the components is 100%.”

183. Xiao also discloses a prior art magnesium alloy represented by Mg-Al-Zn (with AZ91D magnesium alloy being the most widely used) wherein the “main components of this alloy and the respective weight percentages are as follows: **Al 8.3 to 9.7, Zn 0.35 to 1.0, Mn 0.15 to 0.5**, and the remainder is magnesium.” *Id. at ¶ 0003*. Additionally, Xiao discloses a comparative example using AZ91D as a comparative alloy, wherein the composition of the AZ91D comparative alloy is **9 wt % Al, 1 wt % Zn, 0.3 wt % Zr, and 0.1 wt % Mn**, with the remainder being magnesium. *Id. at ¶ 0032*.

184. Further, Xiao discloses that a primary difference between this prior art magnesium alloy and the novel magnesium alloys taught by Xiao’s disclosure lies in the addition of metallic elements like Iron, Copper, and/or Nickel into the alloy. In particular, Xiao discloses that “[e]lements such as Fe, Cu, Ni, Ag, etc., in the magnesium alloy can form a large number of intermetallic composite micro-particles, which can improve the corrosion performance of the magnesium alloy, thereby promoting the decomposition of the magnesium alloy.” *Id. at ¶ 0026, ll. 15–18*. Also comparing the constituents of the comparative example AZ91D magnesium alloy with the other examples in Table 1 of Xiao (Xiao at ¶ 0061), the introduction of Iron, Copper, and/or Nickel (alongside the Aluminum, Zinc, Zirconium, and Manganese already found in the prior art) appears to make the surprisingly large improvement in dissolution rates disclosed by Xiao.

Attorney Docket No. 2189.006

Patent 10,329,653

185. Thus, Xiao discloses a novel magnesium alloy including at least two metals, *i.e.*, zinc and zirconium, at weight percentages within the respective claimed ranges of this claim element of the ‘653 Patent. Xiao also discloses two examples of a prior art magnesium alloy, AZ91D that includes at least four metals, *i.e.*, aluminum, zinc, zirconium, and manganese within the respectively claimed weight percentage ranges of this claim element of the ‘653 Patent. Accordingly, Xiao discloses all of the limitations of this claim element.

- e.       **“and [said dissolvable magnesium alloy composite comprising.. .] about 0.05-45 wt % of a secondary metal [. . . said secondary metal including one or more metals selected from the group consisting of copper, nickel, and cobalt]”**

186. Claim 1 of Xiao discloses a “magnesium alloy, comprising the components at the weight percentages as follows: Al: 13 to 25%, Zn: 2 to 15%, the remainder is Mg, and a sum of the weight percentages of the components is 100%.” Xiao at Claim 1. And Claim 2 of Xiao discloses a “magnesium alloy according to Claim 1, further comprising the trace elements at the weight percentages as follows: **Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%**, Zr: 0.05 to 0.5%, **Ti: 0.05 to 0.5%**; and a sum of the weight percentages of the components is 100%.” Thus, Xiao teaches four candidates to serve as the secondary metal within the claimed weight percentage [and specific metal] limitations of this claim: Copper (Cu), Nickel (Ni), Titanium (Ti), and Iron (Fe). Moreover, as this claim internally defines “a secondary metal” as “including one or more metals,” Xiao also discloses a range of secondary metal running **from 0.05 wt %** (*i.e.*, the low end of the Copper, Nickel or Titanium ranges) **to 15.5 wt %** (*i.e.*, the accumulation of the high end of the Copper, Nickel, Titanium, and Iron ranges (5 wt % + 5 wt % + 0.5 wt% + 5 wt%)). Accordingly, Xiao teaches this limitation of the claim.

Attorney Docket No. 2189.006

Patent 10,329,653

- f. “[a secondary metal] to form a galvanically-active intermetallic particle that promotes corrosion of said dissolvable magnesium alloy composite,”

187. Xiao discloses that its invention it adopts a magnesium alloy with high aluminum and zinc content and further adds elements Fe, Cu, Ni and Ag which can form intermetallics and enhance the corrosion performance of the magnesium alloy. Xiao explains the corrosion mechanism within a magnesium alloy in terms of a cathode phase, which Xiao explains includes “the  $\beta$  ( $Mg_{17}Al_{12}$ ) phase and eutectic phase on the grain boundary of the magnesium alloy,” and “the magnesium matrix  $\alpha$  phase which functions as an anode phase, so that the matrix and the grain boundary of the magnesium alloy form a large amount of micro-batteries, which [micro-batteries] greatly accelerate the corrosion decomposition of magnesium alloy.” *Id.* at ¶ 0026.

188. Xiao discloses that “[e]lements such as Fe, Cu, Ni, Ag, etc., in the magnesium alloy can form a large number of intermetallic composite micro-particles, which can improve the corrosion performance of the magnesium alloy, thereby promoting the decomposition of the magnesium alloy.” *Id.* at ¶ 0026. These elements are among the claimed “secondary metals” disclosed by Xiao. Thus, all of the limitations of this claim element are disclosed by Xiao.

- g. “said secondary metal including one or more metals selected from the group consisting of copper, nickel, and cobalt,”

189. Claim 2 of Xiao discloses a “magnesium alloy according to Claim 1, further comprising the trace elements at the weight percentages as follows: Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%; and a sum of the weight percentages of the components is 100%.” Of the additive materials taught in Example 7 of Xiao (i.e. Al-Fe, Al-Ni, Al-Ag, Al-Cu, Al-Zr, Al-Ti and Zinc), Al-Ni, and Al-Cu contain copper and nickel. This

Attorney Docket No. 2189.006

Patent 10,329,653

claim element only requires the secondary metal include one of these metals. Thus, Xiao teaches two candidates to serve as the secondary metal of this claim: Copper (Cu) and Nickel (Ni).

**h. “said magnesium alloy composite has a dissolution rate of at least 5 mg/cm<sup>2</sup>/hr. in 3 wt % KCl water mixture at 90°C.”**

190. A POSITA in April 2014 would have understood the English translation of Xiao to disclose a table of dissolution rates (“decomposition rates”) because the units of measurement are the units used by POSITAs to measure dissolution rates. In particular, Xiao provides dissolution rates for its various examples, as follows:

	Room temperature tensile strength $\sigma_b$ (MPa)	Decomposition rate at 70 °C in 3% KCl solution (g.cm <sup>-2</sup> .h <sup>-1</sup> )	Decomposition rate at 93 °C in 3% KCl solution (g.cm <sup>-2</sup> .h <sup>-1</sup> )
Comparative example 1	232	0.00026	0.0005
Example 1	360	0.035	0.074
Example 2	385	0.015	0.045
Example 3	410	0.013	0.036
Example 4	375	0.034	0.058
Example 5	392	0.025	0.048
Example 6	365	0.021	0.063
Example 7	387	0.036	0.057

Xiao at ¶ 0064.

191. Xiao provides dissolution rates in g/cm<sup>2</sup>/hr, while the claims of the '653 Patent express dissolution rates in mg/cm<sup>2</sup>/hr. A POSITA in April 2014 would have known that converting from g/cm<sup>2</sup>/hr to mg/cm<sup>2</sup>/hr, merely requires multiplication of the numbers disclosed

Attorney Docket No. 2189.006

Patent 10,329,653

in this table in Xiao by 1,000.

192. Thus, the dissolution rates disclosed in Xiao convert to:

	Decomposition rate at 70°C in 3% KCl solution mg/cm <sup>2</sup> /hr	Decomposition rate at 93°C in 3% KCl solution mg/cm <sup>2</sup> /hr
Comparative example 1	0.26	0.5
Example 1	35	74
Example 2	15	45
Example 3	13	36
Example 4	34	58
Example 5	25	48
Example 6	21	63
Example 7	36	57

193. Each of the seven examples (excluding the comparative [prior art] example) provided in Xiao therefore discloses a dissolution rate of “at least 5 mg/cm<sup>2</sup>/hr in 3 wt.% KCl water” at both 70°C or 93°C. Given that the disclosed dissolution rates of Xiao both above and below 90°C are well above the claimed floor of “at least 5 mg/cm<sup>2</sup>/hr” (i.e. at least three times overall and for Example 7 at least seven times) it is my opinion that a POSITA in April 2014 would have recognized the dissolution rate of the examples in Xiao at 93°C to also be necessarily well

Attorney Docket No. 2189.006

Patent 10,329,653

above the claimed floor of “at least of 5 mg/cm<sup>2</sup>/hr.” Consequently, this element of Claim 1 is also disclosed by Xiao.

194. Accordingly, as set forth in the immediately foregoing paragraphs, it is my opinion that Xiao would have fully disclosed each and every element of Claim 41 of the ‘653 Patent to a POSITA in or before April 2014 in accordance with Patent Owner’s definitions (set forth in Paragraph 78 of my Declaration).

## 8. Dependent Claim 43

- a. **“The dissolvable magnesium composite as defined in claim 41, wherein said secondary metal includes nickel,”**

195. As set forth above, it is my opinion that Xiao would have disclosed all elements of Claim 41 to a POSITA in April 2014.

196. Claim 2 of Xiao discloses a “magnesium alloy according to claim 1, further comprising trace elements at the weight percentages as follows: Fe: 0.1 to 5%, Cu: 0.05 to 5%, **Ni: 0.05 to 5%**, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%; and a sum of the weight percentages of the components is 100%.” Thus, Xiao discloses the use of nickel as a secondary metal.

- b. **“said nickel forms galvanically-active in situ precipitate in said magnesium composite.”**

197. Xiao discloses that “[e]lements such as Fe, Cu, **Ni**, Ag, etc., in the magnesium alloy can form a large number of **intermetallic composite micro-particles**, which can improve the corrosion performance of the magnesium alloy, thereby promoting the decomposition of the magnesium alloy.” Xiao at ¶ 0026.

198. A POSITA in April 2014 would have understood that a precipitate would necessarily be formed in place and that the “composite microparticles” disclosed by Xiao are the same as the claimed “precipitate” (i.e. “something having a new phase different from what existed

Attorney Docket No. 2189.006

Patent 10,329,653

before” under Patent Owner’s proposed definition). Consequently, all of the limitations of Dependent Claim 43 are taught by Xiao.

## 9. Independent Claim 45

### a. “A dissolvable magnesium alloy composite”

199. Xiao discloses “a **magnesium alloy**...and further adds elements of **Fe, Cu, Ni, and Ag** which can **enhance the corrosion performance** of the magnesium alloy...” Xiao at ¶ 0026, ll. 1–9.

200. Thus, Xiao’s disclosure of a combination of a magnesium alloy and elements of iron, copper, nickel, and silver thus meets the construction of “a dissolvable magnesium composite.”

### b. “for use in a **ball or other tool component** in a well drilling or completion operation,”

201. Xiao discloses that the cast magnesium alloy “**can be used as a tripping ball material for a multi-stage sliding sleeve staged-fracturing technique.**” *Id.* at ¶ 0001. A POSITA in August 2014 would have understood that a tripping ball was a type of tool component in an oil well drilling operation. Thus, Xiao teaches this limitation of the claim.

### c. “**said dissolvable magnesium alloy composite comprising over 50 % magnesium;**”

202. Claim 1 of Xiao discloses a “magnesium alloy, comprising the components at the weight percentages as follows: **Al: 13 to 25%, Zn: 2 to 15%, the remainder is Mg,** and a sum of the weight percentages of the components is 100%.” As such, the total range of the non-magnesium elements disclosed in Claim 1 of Xiao is 15% (*i.e.*, 13 + 2) to 40% (*i.e.*, 25 + 15), leaving **85 to 60 wt%** for the **magnesium**. The Examples of Xiao disclose a range of magnesium **from 54.5 wt%**

Attorney Docket No. 2189.006

Patent 10,329,653

- **79.2 wt%.** Thus, Xiao discloses a range of magnesium of 54.5 wt% to 85 wt %, which is over 50 wt % magnesium, thus fully disclosing this element of the claim.

- d. “[said dissolvable magnesium alloy composite comprising . . .] one or more metals selected from the group consisting of **0.1-3 wt % zinc, 0.01-1 wt % zirconium, 0.05-1 wt % manganese, 0.0002-0.04 wt % boron, and 0.4-0.7 wt % bismuth;”**

203. Claim 1 of Xiao discloses a “magnesium alloy, comprising the components at the weight percentages as follows: Al: 13 to 25%, **Zn: 2 to 15%,**” and Claim 2 of Xiao discloses secondary metals of “Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%, **Zr: 0.05 to 0.5%,** Ti: 0.05 to 0.5%; and a sum of the weight percentages of the components is 100%.”

204. Thus, even though this claim element only requires certain weight percentages of one of zinc, zirconium, manganese, boron or bismuth, Xiao actually discloses a dissolvable magnesium alloy composite including weight ranges of both zinc (Zn) and zirconium (Zr), which overlap a portion of the ranges required by this claim element for those metallic elements. Accordingly, Xiao teaches this limitation of the claim.

- e. “[and [said dissolvable magnesium alloy composite comprising] **about 0.05-45 wt % of a secondary metal** [. . . said secondary metal including one or more metals selected from the group consisting of copper, nickel, and cobalt]”]

205. Claim 2 of Xiao discloses a “magnesium alloy according to Claim 1, further comprising the trace elements at the weight percentages as follows: **Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%,** Zr: 0.05 to 0.5%, **Ti: 0.05 to 0.5%;** and a sum of the weight percentages of the components is 100%.” Thus, Xiao teaches four candidates to serve as the secondary metal within the claimed weight percentage [and specific metal] limitations of this claim: **Copper (Cu), Nickel (Ni), Titanium (Ti), and Iron (Fe).** Moreover, as this claim internally defines “a secondary metal” as “including one or more metals,” Xiao also discloses a range of secondary metal running

Attorney Docket No. 2189.006

Patent 10,329,653

**from 0.05 wt %** (i.e., the low end of the Copper and Nickel ranges) **to 10 wt %** (i.e., the accumulation of the high end of the Copper and Nickel ranges (5 wt % + 5 wt %)). Accordingly, Xiao meets this element of the claim.

- f.       **“[a secondary metal] to form a galvanically-active intermetallic particle that promotes corrosion of said dissolvable magnesium alloy composite,”**

206. Xiao discloses that its invention it adopts a magnesium alloy with high aluminum and zinc content and **further adds elements Fe, Cu, Ni and Ag which can form intermetallics** and **enhance the corrosion performance of the magnesium alloy**. Xiao explains the corrosion mechanism within a magnesium alloy in terms of a cathode phase, which Xiao explains includes “the  $\beta$  ( $Mg_{17}Al_{12}$ ) phase and eutectic phase on the grain boundary of the magnesium alloy,” and “the magnesium matrix  $\alpha$  phase which functions as an anode phase, so **that the matrix and the grain boundary of the magnesium alloy form a large amount of micro-batteries, which [micro-batteries] greatly accelerate the corrosion decomposition of magnesium alloy.**” *Id.* at ¶ 0026.

207. Xiao discloses that “[e]lements such as Fe, Cu, Ni, Ag, etc., in the magnesium alloy **can form a large number of intermetallic composite micro-particles**, which can **improve the corrosion performance of the magnesium alloy, thereby promoting the decomposition of the magnesium alloy.**” *Id.* at ¶ 0026. These elements are among the claimed “secondary metals” disclosed by Xiao. Thus, all of the limitations of this claim element are disclosed by Xiao.

- g.       **“said secondary metal including one or more metals selected from the group consisting of copper, nickel, and cobalt,”**

208. Claim 2 of Xiao discloses a “magnesium alloy according to Claim 1, further comprising the trace elements at the weight percentages as follows: Fe: 0.1 to 5%, **Cu: 0.05 to 5%, Ni: 0.05 to 5%**, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%; and a sum of the weight percentages of

Attorney Docket No. 2189.006

Patent 10,329,653

the components is 100%.” Of the additive materials taught in Example 7 of Xiao (i.e. Al-Fe, Al-Ni, Al-Ag, Al-Cu, Al-Zr, Al-Ti and Zinc), **Al-Ni**, and **Al-Cu** contain **copper and nickel**. This claim element only requires the secondary metal include one of these metals. Thus, Xiao teaches two candidates to serve as the secondary metal of this claim: Copper (Cu) and Nickel (Ni).

**h. “said magnesium alloy composite has a dissolution rate of at least 5 mg/cm<sup>2</sup>/hr. in 3 wt % KCl water mixture at 90°C.”**

209. A POSITA in April 2014 would have understood the English translation of Xiao to disclose a table of dissolution rates (“decomposition rates”) because the units of measurement are the units used by POSITAs to measure dissolution rates. In particular, Xiao provides dissolution rates for its various examples, as follows:

	Room temperature tensile strength $\sigma_b$ (MPa)	Decomposition rate at 70 °C in 3% KCl solution (g.cm <sup>-2</sup> .h <sup>-1</sup> )	Decomposition rate at 93 °C in 3% KCl solution (g.cm <sup>-2</sup> .h <sup>-1</sup> )
Comparative example 1	232	0.00026	0.0005
Example 1	360	0.035	0.074
Example 2	385	0.015	0.045
Example 3	410	0.013	0.036
Example 4	375	0.034	0.058
Example 5	392	0.025	0.048
Example 6	365	0.021	0.063
Example 7	387	0.036	0.057

Xiao at ¶ 0064.

210. Xiao provides dissolution rates in g/cm<sup>2</sup>/hr, while the claims of the '653 Patent

Attorney Docket No. 2189.006

Patent 10,329,653

express dissolution rates in mg/cm<sup>2</sup>/hr. A POSITA in April 2014 would have known that converting from g/cm<sup>2</sup>/hr to mg/cm<sup>2</sup>/hr, merely requires multiplication of the numbers disclosed in this table in Xiao by 1,000.

211. Thus, the dissolution rates disclosed in Xiao convert to:

	Decomposition rate at 70°C in 3% KCl solution mg/cm <sup>2</sup> /hr	Decomposition rate at 93°C in 3% KCl solution mg/cm <sup>2</sup> /hr
Comparative example 1	0.26	0.5
Example 1	35	74
Example 2	15	45
Example 3	13	36
Example 4	34	58
Example 5	25	48
Example 6	21	63
Example 7	36	57

212. Each of the seven examples (excluding the comparative [prior art] example) provided in Xiao therefore discloses a dissolution rate of “at least 5 mg/cm<sup>2</sup>/hr in 3 wt.% KCl water” at both 70°C or 93°C. Given that the disclosed dissolution rates of Xiao both above and below 90°C are well above the claimed floor of “at least 5 mg/cm<sup>2</sup>/hr” (i.e. at least three times overall and for Example 7 at least seven times) it is my opinion that a POSITA in April 2014 would have recognized the dissolution rate of the examples in Xiao at 93°C to also be necessarily well above the claimed floor of “at least of 5 mg/cm<sup>2</sup>/hr.” Consequently, this element of Claim 1 is also disclosed by Xiao.

Attorney Docket No. 2189.006

Patent 10,329,653

213. Accordingly, as set forth in the immediately foregoing paragraphs, it is my opinion that Xiao would have fully disclosed each and every element of Claim 45 of the ‘653 Patent to a POSITA in or before April 2014 in accordance with Patent Owner’s definitions (set forth in Paragraph 78 of my Declaration).

## **10. Independent Claim 49**

### **a. “A magnesium composite”**

214. Xiao discloses a “**magnesium alloy**...and further adds elements of Fe, Cu, Ni, and Ag which can enhance the corrosion performance of the magnesium alloy...” Xiao at ¶ 0026, ll. 1–9.

215. Thus, Xiao’s disclosure of a combination of a magnesium alloy and elements of iron, copper, nickel, and silver thus meets the construction of “a magnesium composite.”

### **b. “[magnesium composite] that includes in situ precipitation of galvanically-active intermetallic phases to enable controlled dissolution of said magnesium composite.”**

216. Xiao discloses that its invention it adopts a magnesium alloy with high aluminum and zinc content and **further adds elements Fe, Cu, Ni and Ag which can form intermetallics** and **enhance the corrosion performance of the magnesium alloy**. Xiao explains the corrosion mechanism within a magnesium alloy in terms of a cathode phase, which Xiao explains includes “the  $\beta$  ( $Mg_{17}Al_{12}$ ) phase and eutectic phase on the grain boundary of the magnesium alloy,” and “the magnesium matrix  $\alpha$  phase which functions as an anode phase, so **that the matrix and the grain boundary of the magnesium alloy form a large amount of micro-batteries, which [micro-batteries] greatly accelerate the corrosion decomposition of magnesium alloy.**” *Id.* at ¶ 0026.

Attorney Docket No. 2189.006

Patent 10,329,653

217. Xiao further discloses that “[e]lements such as Fe, Cu, Ni, Ag, etc., in the magnesium alloy **can form a large number of intermetallic composite micro-particles**, which can improve the corrosion performance of the magnesium alloy, thereby promoting the decomposition of the magnesium alloy.” Xiao at ¶ 0026, ll. 15–18. A POSITA in August 2014 would have understood this disclosure of “intermetallic composite micro-particles” to be a reference to intermetallic precipitate.

218. A POSITA reading the Xiao reference in April 2014 would have recognized that the precipitation of galvanically-active intermetallic phases (“micro-batteries”) are naturally and necessarily generated by the *in situ* reaction between magnesium and any and all of the disclosed metal additives forming *in situ* precipitate. Thus, all of the limitations of this claim element are disclosed by Xiao.

c. **“said magnesium composite comprising a mixture of magnesium or a magnesium alloy and an additive material,”**

219. Xiao discloses “first loading **pure magnesium and pure aluminum** into a smelting furnace . . . , **next loading** pure zinc and **an [sic] intermediate alloys** of trace element components into a **resulting magnesium-aluminum alloy melt** after melting.” *Id.* at ¶ 0022.

220. Xiao discloses that these “intermediate alloys” are **Al-Fe, Al-Ni, Al-Cu, Al-Ag, Al-Zr, Al-Ti**, which are heated to dry and then added to the magnesium-aluminum alloy melt.” Xiao at ¶ 0023. These intermediate alloys meet Patent Owner’s proposed definition of “additive material,” i.e. “a material that is added.”

221. The addition of one or more of these intermediate alloys to the melted magnesium alloy necessarily results in a mixture of magnesium or magnesium alloy and the additive material. Thus, Xiao discloses all of the limitations of this claim element.

Attorney Docket No. 2189.006

Patent 10,329,653

- d. “said **additive material** constituting **about 0.05-45 wt %** of said mixture, [said additive material includes one or more metals selected from the group consisting of copper, nickel, titanium, iron, and cobalt]”

222. Claim 1 of Xiao discloses: a “magnesium alloy, comprising the components at the weight percentages as follows: **Al: 13 to 25%, Zn: 2 to 15%**, the remainder is Mg, and a sum of the weight percentages of the components is 100%.” Xiao at Claim 1. And Claim 2 discloses: a “magnesium alloy, according to Claim 1, further comprising the trace elements at the weight percentages as follows: **Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%**, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%; and a sum of the weight percentages of the components is 100%.” *Id.* at Claim 2.

223. These metallic elements are found in the additive materials generally disclosed by Xiao (*i.e.*, **intermediate alloys: Al-Fe, Al-Ni, Al-Cu, Al-Ti**) (Xiao at ¶ 0023) and particularly described in association with Example 7 of Xiao (*Id.* at ¶¶ 0057 - 0060) for which I have already calculated (and set forth above) the specific weight percentage (ranges) of each additive material (which has been defined by the Patent Owner as “a material that is added” (see Paragraph 78 of my Declaration)) set forth in Example 7 of Xiao (*i.e.* Al-Fe, Al-Ni, Al-Ag, Al-Cu, Al-Zr, Al-Ti and Zinc).

224. In particular, the additive materials aluminum-iron with 30 wt% iron, aluminum-nickel with 45 wt% nickel, and aluminum-copper with 40 wt% copper, which each have greater melting point temperatures than the solidus temperature of magnesium or the magnesium aluminum alloy disclosed in Xiao, also fall well within the range of about 0.05 wt% to 45 wt% of the mixture. For example, the **aluminum-iron** additive constitutes 3.3 wt% of the mixture (*i.e.*, 1%/0.30); the **aluminum-nickel** additive constitutes 1.1 wt% of the mixture (*i.e.*, 0.5%/0.45); and the **aluminum-copper** additive constitutes 0.25 wt% of the mixture (*i.e.*, 0.1%/0.40). A POSITA in April 2014 would have also recognized that the accumulated weight percentages of all of the

Attorney Docket No. 2189.006

Patent 10,329,653

secondary metals in Example 7 of Xiao (using the alloy compositions discussed above) would have been 9.65 wt% (*i.e.*, 3.3 wt% + 1.1 wt% + 0.25 wt% + 5.0 wt%), also within the claimed range. Thus, Xiao teaches secondary metals having 0.05 to 15.5 wt%. Accordingly, Xiao teaches this limitation of the claim.

- e. “said **additive material** includes one or more metals selected from the group consisting of copper, nickel, titanium, iron, and cobalt,”

225. Xiao discloses one or more of the “secondary metals,” including **copper (Cu), nickel (Ni), titanium (Ti) and iron (Fe)** as follows: “**Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%.**” Xiao at Claim 2. Of the additive materials taught in Example 7 of Xiao (*i.e.* Al-Fe, Al-Ni, Al-Ag, Al-Cu, Al-Zr, Al-Ti and Zinc), **Al-Fe, Al-Ni, Al-Cu, and Al-Ti** contain **copper, nickel, titanium and iron**. This claim element only requires the secondary metal include one of these metals. Thus, Xiao teaches four candidates to serve as the secondary metal of this claim: Copper (Cu), Nickel (Ni), Titanium (Ti), and Iron (Fe). Accordingly, all of the elements of this claim limitation are disclosed by Xiao.

- f. “said magnesium composite including in situ precipitation of galvanically-active intermetallic phases **that include [sic] said additive material**,”

226. Xiao discloses that its invention it adopts a magnesium alloy with high aluminum and zinc content and **further adds elements Fe, Cu, Ni and Ag which can form intermetallics** and enhance the corrosion performance of the magnesium alloy. Xiao explains the corrosion mechanism within a magnesium alloy in terms of a cathode phase, which Xiao explains includes “the  $\beta$  ( $Mg_{17}Al_{12}$ ) phase and eutectic phase on the grain boundary of the magnesium alloy,” and “the magnesium matrix  $\alpha$  phase which functions as an anode phase, so **that the matrix and the grain boundary of the magnesium alloy form a large amount of micro-batteries, which**

Attorney Docket No. 2189.006

Patent 10,329,653

[micro-batteries] greatly accelerate the corrosion decomposition of magnesium alloy.” *Id.* at ¶ 0026.

227. Xiao further discloses that “[e]lements such as **Fe, Cu, Ni, Ag**, etc., in the magnesium alloy **can form a large number of intermetallic composite micro-particles**, which can improve the corrosion performance of the magnesium alloy, thereby promoting the decomposition of the magnesium alloy.” *Id.* at ¶ 0026, ll. 15–18. A POSITA in August 2014 would have understood this disclosure of “intermetallic composite micro-particles” to be a reference to intermetallic precipitate. As noted above, at least Fe, Cu, and Ni are “additive materials.” Consequently, the in situ precipitation of galvanically-active intermetallic phases will include one or more of these additive materials. Thus, Xiao discloses all of this element of this claim limitation.

g.     **“said additive material located in sufficient quantities in said galvanically-active intermetallic phases so as to obtain a composition and morphology of said galvanically-active intermetallic phases such that a galvanic corrosion rate along said galvanically-active intermetallic phases causes said magnesium composite to have a dissolution rate of at least at least 5 mg/cm<sup>2</sup>/hr. in 3 wt % KCl water mixture at 90°C.”**

228. Claim 2 of Xiao discloses: a “magnesium alloy, according to claim 1, further comprising the trace elements at the weight percentages as follows: **Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%**, Zr: 0.05 to 0.5%, **Ti: 0.05 to 0.5%**; and a sum of the weight percentages of the components is 100%.” *Id.* at Claim 2. Thus, Xiao discloses four metals that could serve as the claimed “additive material” and have weight % quantities that fall within the range of this claim—i.e, they are located in “sufficient quantities.”

229. Xiao discloses that “[e]lements such as Fe, Cu, Ni, Ag, etc., in the magnesium alloy can form a large number of intermetallic composite micro-particles, which can improve the corrosion performance of the magnesium alloy, thereby promoting the decomposition of the

Attorney Docket No. 2189.006

Patent 10,329,653

magnesium alloy.” *Id.* ¶ 0026. A POSITA would understand that the galvanically-active intermetallic phases (“composite micro-particles”) have a morphology that enhances the corrosion dissolution rate of the alloy due to a larger number of smaller particles throughout the microstructure. A POSITA would also understand the relationship between “composite micro-particles” and morphology of the galvanically-active intermetallic phases.

230. A POSITA in April 2014 would have understood the English translation of Xiao to disclose a table of dissolution rates (“decomposition rates”) because the units of measurement in that table are units regularly used by POSITAs to express measurements of dissolution. In particular, Xiao provides dissolution rates for its various examples, as follows:

	Room temperature tensile strength $\sigma_b$ (MPa)	Decomposition rate at 70 °C in 3% KCl solution (g.cm <sup>-2</sup> .h <sup>-1</sup> )	Decomposition rate at 93 °C in 3% KCl solution (g.cm <sup>-2</sup> .h <sup>-1</sup> )
Comparative example 1	232	0.00026	0.0005
Example 1	360	0.035	0.074
Example 2	385	0.015	0.045
Example 3	410	0.013	0.036
Example 4	375	0.034	0.058
Example 5	392	0.025	0.048
Example 6	365	0.021	0.063
Example 7	387	0.036	0.057

Xiao at ¶ 0064.

231. Xiao provides dissolution rates in g/cm<sup>2</sup>/hr, while the claims of the '653 Patent

Attorney Docket No. 2189.006

Patent 10,329,653

express the claimed dissolution rate in mg/cm<sup>2</sup>/hr. A POSITA in April 2014 would have known that converting from g/cm<sup>2</sup>/hr to mg/cm<sup>2</sup>/hr, merely requires multiplication of the numbers disclosed in this table in Xiao by 1,000.

**2.** Thus, the dissolution rates disclosed in Xiao convert to:

	Decomposition rate at 70°C in 3% KCl solution mg/cm <sup>2</sup> /hr	Decomposition rate at 93°C in 3% KCl solution mg/cm <sup>2</sup> /hr
Comparative example 1	0.26	0.5
Example 1	35	74
Example 2	15	45
Example 3	13	36
Example 4	34	58
Example 5	25	48
Example 6	21	63
Example 7	36	57

232. Each of the seven examples (excluding the comparative [prior art] example) provided in Xiao therefore discloses a dissolution rate of “at least 5 mg/cm<sup>2</sup>/hr in 3 wt.% KCl water” at both 70°C or 93°C. Given that the disclosed dissolution rates of Xiao both above and below 90°C are well above the claimed floor of “at least 5 mg/cm<sup>2</sup>/hr” (i.e. at least three times overall and for Example 7 at least seven times) it is my opinion that a POSITA in April 2014 would have recognized the dissolution rate of the examples in Xiao at 93°C to also be necessarily well above the claimed floor of “at least of 5 mg/cm<sup>2</sup>/hr.” Consequently, this element of Claim 1 is also disclosed by Xiao.

Attorney Docket No. 2189.006

Patent 10,329,653

233. Accordingly, as set forth in the immediately foregoing paragraphs, it is my opinion that Xiao would have fully disclosed each and every element of Claim 49 of the ‘653 Patent to a POSITA in or before April 2014 in accordance with Patent Owner’s definitions (set forth in Paragraph 78 of my Declaration).

### **11. Dependent Claim 69**

- a. “The magnesium composite as defined in claim 49, wherein said additive material has a melting point temperature that is 100°C. greater than a melting temperature of said magnesium or magnesium alloy.”

234. As set forth above, it is my opinion that Xiao would have disclosed all elements of Claim 49 to a POSITA in April 2014.

235. A POSITA would have understood “melting temperature” in this claim element to be a reference to the smelting temperature or temperature of the melt when the additive material is being added to the magnesium or magnesium alloy, not the “melting point temperature” as that measurement is used for the additive material.

236. Xiao generally discloses “loading pure magnesium and pure aluminum into a smelting furnace and increasing the temperature to 700 to 730 °C, next loading pure zinc and an [sic] intermediate alloys of trace element components into a resulting magnesium-aluminum alloy melt after melting and increasing the temperature to 740 to 780°C . . . .” Xiao at ¶¶ 0021–0022; see also *Id.* at Claim 5 (“the melting temperature of the pure magnesium and pure aluminum is from 700 to 730 °C.”)

237. Examples 1-7 of Xiao teach different melting or smelting temperatures for the magnesium-aluminum alloy, ranging from a low of 700°C in Example 1 to a high of 730 °C in Examples 2, 5, and 7. Xiao ¶¶ 0034–0060. In each Example, “[a]fter melting” the intermediate

Attorney Docket No. 2189.006

Patent 10,329,653

alloy are “added into the resulting magnesium-aluminum alloy melt, and at the same time the temperature is raised” to a different temperature depending on the example. *Id.*

238. The **additive materials** noted above are one or more of the **intermediate alloys:** **Al-Fe, Al-Ni, Al-Cu, Al-Ag, Al-Zr, Al-Ti.** *Id.* at ¶ 0023. As I noted in Paragraph 78 of Declaration, Patent Owner has taken the position that “melting point temperature” would have meant “temperature at which liquid is formed” to a POSITA in April 2014.

239. As already noted in Paragraph 152 of my Declaration (above), based on the typical phase diagrams, a POSITA in April 2014 would have recognized that **Al-Fe** had a variable melting point temperature ranging from 1538°C (pure iron) down to 652°C for a eutectic point near 99 wt% aluminum; **Al-Ni** had a variable melting point temperature that ranges from 1640°C down to 640°C (depending upon the alloy mixture); and **Al-Cu** had a variable melting point temperature ranging from 548°C up to 1084°C (depending upon the alloy mixture). A POSITA in April 2014 would have been able to determine the specific melting point temperature of a specific alloy mixture based on the content of these typical phase diagrams.

240. Example 7 specifically discloses a melting or smelting temperature of 730°C. Xiao at ¶ 0060. I specifically analyzed Example 7 of Xiao above to determine (using particular intermediate alloy compositions) the melting point temperatures of the intermediate alloys added. As a result, it is my opinion that a POSITA in April 2014 would recognize from Example 7 of Xiao that at least an **aluminum-nickel** additive having 45 wt% nickel has a melting point temperature of 854°C which is 100°C greater than the melting or smelting temperatures of the magnesium aluminum alloy used in Example 7 (*i.e.*, 730°C) and otherwise taught generally by Xiao (*i.e.*, 700-730°C). A POSITA in April 2014 would have also recognized that other intermediate alloy compositions could have fallen within the teachings of Example 7 of Xiao as

Attorney Docket No. 2189.006

Patent 10,329,653

well as the other teachings of Xiao and resulted in **Al-Fe, Al-Ni, and Al-Cu intermediate alloys** that would have had melting point temperatures that were 100°C. greater than the smelting temperatures of the magnesium alloy generally taught by Xiao (*i.e.*, 700-730°C).<sup>14</sup> Thus, Xiao discloses all of the limitations of Dependent Claim 69.

## 12. Dependent Claim 70

- a.     **“The magnesium composite as defined in claim 49, wherein said magnesium composite is at least partially included in a down hole well component,”**

241. As set forth above, it is my opinion that Xiao would have disclosed all elements of Claim 49 to a POSITA in April 2014.

242. Xiao discloses that the cast magnesium alloy “**can be used as a tripping ball material for a multi-stage sliding sleeve staged-fracturing technique.**” *Id.* at ¶ 0001. A POSITA in August 2014 would have understood that a tripping ball was a type of tool component in an oil well drilling operation. Thus, Xiao teaches this limitation of the claim.

- b.     **“said down hole well component including one or more components selected from the group consisting of a sleeve, frac**

---

<sup>14</sup> It is worth pointing out—although I would disagree with either notion—that if Patent Owner argues either that “melting temperature” was a typographical error (which I doubt given the other claims that say “melting point temperature” and the numerous Certificates of Correction that did not address this issue) or simply argues that “melting temperature of said magnesium or magnesium alloy” means “melting point temperature of said magnesium or magnesium alloy,” not only would the Al-Ni intermediate alloy with 45 wt% nickel exceed the **melting point temperature** of pure magnesium (*i.e.*, 650°C) and the **melting point temperature of the initially loaded magnesium-aluminum alloy** (*i.e.*, 438°C) of Example 7 using the constituents discussed in Paragraphs 114-118 of my Declaration (*i.e.*, a composition of 78.5 wt% Mg and 21.5 wt% Al), by at least 100°C, but also the Al-Cu, Al-Fe, Al-Ti and Al-Zr intermediate alloys, having the constituents set forth in those Paragraphs of my Declaration would similarly exceed that limitation.

Attorney Docket No. 2189.006

Patent 10,329,653

**ball, hydraulic actuating tooling, tube, valve, valve component, or plug.”**

243. Xiao discloses that the cast magnesium alloy “**can be used as a tripping ball material for a multi-stage sliding sleeve staged-fracturing technique.**” *Id.* at ¶ 0001. A POSITA in August 2014 would have understood that a tripping ball was a type of tool component in an oil well drilling operation. Thus, Xiao teaches this limitation of the claim. Consequently, all of the limitations of Dependent Claim 70 are taught by Xiao.

### **13. Independent Claim 73**

#### **a. “A dissolvable magnesium composite”**

244. Xiao discloses “a **magnesium alloy**...and further adds elements of **Fe, Cu, Ni, and Ag** which can **enhance the corrosion performance** of the magnesium alloy...” Xiao at ¶ 0026, ll. 1–9.

245. Thus, Xiao’s disclosure of a combination of a magnesium alloy and elements of iron, copper, nickel, and silver thus meets the construction of “a dissolvable magnesium composite.”

#### **b. “for use in a ball or other tool component in a well drilling or completion operation,”**

246. Xiao discloses that the cast magnesium alloy “**can be used as a tripping ball material for a multi-stage sliding sleeve staged-fracturing technique.**” *Id.* at ¶ 0001. A POSITA in August 2014 would have understood that a tripping ball was a type of tool component in an oil well drilling operation. Thus, Xiao teaches this limitation of the claim.

Attorney Docket No. 2189.006

Patent 10,329,653

- c. “**said dissolvable magnesium composite includes in situ precipitation of galvanically-active intermetallic phases to enable controlled dissolution of said magnesium composite,**”

247. Xiao discloses that its invention it adopts a magnesium alloy with high aluminum and zinc content and **further adds elements Fe, Cu, Ni and Ag which can form intermetallics** and **enhance the corrosion performance of the magnesium alloy.** Xiao explains the corrosion mechanism within a magnesium alloy in terms of a cathode phase, which Xiao explains includes “the  $\beta$  ( $Mg_{17}Al_{12}$ ) phase and eutectic phase on the grain boundary of the magnesium alloy,” and “the magnesium matrix  $\alpha$  phase which functions as an anode phase, so **that the matrix and the grain boundary of the magnesium alloy form a large amount of micro-batteries, which [micro-batteries] greatly accelerate the corrosion decomposition of magnesium alloy.**” *Id.* at ¶ 0026.

248. Xiao further discloses that “[e]lements such as Fe, Cu, Ni, Ag, etc., in the magnesium alloy **can form a large number of intermetallic composite micro-particles**, which can improve the corrosion performance of the magnesium alloy, thereby promoting the decomposition of the magnesium alloy.” Xiao at ¶ 0026, ll. 15–18. A POSITA in August 2014 would have understood this disclosure of “intermetallic composite micro-particles” to be a reference to intermetallic precipitate.

249. A POSITA reading the Xiao reference in April 2014 would have recognized that the precipitation of galvanically-active intermetallic phases (“micro-batteries”) are naturally and necessarily generated by the *in situ* reaction between magnesium and any and all of the disclosed metal additives forming *in situ* precipitate. Thus, all of the limitations of this claim element are disclosed by Xiao.

Attorney Docket No. 2189.006

Patent 10,329,653

d. “said magnesium composite comprising a mixture of **magnesium or a magnesium alloy** and an **additive material**,”

250. Xiao discloses “first loading **pure magnesium and pure aluminum** into a smelting furnace . . . , **next loading** pure zinc and **an [sic] intermediate alloys** of trace element components into a **resulting magnesium-aluminum alloy melt** after melting.” *Id.* at ¶ 0022.

251. Xiao discloses that these “intermediate alloys” are **Al-Fe, Al-Ni, Al-Cu, Al-Ag, Al-Zr, Al-Ti**, which are heated to dry and then added to the magnesium-aluminum alloy melt.” Xiao at ¶ 0023. These intermediate alloys meet Patent Owner’s proposed definition of “additive material,” i.e. “a material that is added.”

252. The addition of one or more of these intermediate alloys to the melted magnesium alloy necessarily results in a mixture of magnesium or magnesium alloy and the additive material. Thus, Xiao discloses all of the limitations of this claim element.

e. “**said additive material constituting about 0.05 wt % of said mixture, said additive material is a metal or metal alloy,**”

253. The overall teaching of Xiao notes the use of a variety of metals as additive materials. See, e.g., Xiao at ¶ 0026. For instance, Claim 2 of Xiao discloses a “magnesium alloy according to claim 1, further comprising the trace elements at the weight percentages as follows: Fe: 0.1 to 5%, **Cu: 0.05 to 5%, Ni: 0.05 to 5%, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%**; and a sum of the weight percentages of the components is 100%.” *Id.* This disclosure references a number of different metal additive materials (i.e. copper, nickel, zirconium and titanium) each of which Xiao teaches may constitute about 0.05wt % of the mixture. Thus, Xiao fully discloses these limitations of this claim element.

Attorney Docket No. 2189.006

Patent 10,329,653

- f.       “**said additive material includes one or more metals selected from the group consisting of copper, nickel, titanium, iron, silicon, and cobalt,**”

254. Of the additive metal materials discussed in the immediately preceding paragraph (i.e. copper, nickel, zirconium and titanium), each disclosed as one of the potential options among six metals. This claim element requires the additive material include even only one of these metals. Accordingly, Xiao discloses this element of the claim.

- g.       “**said magnesium composite including in situ precipitation of galvanically-active intermetallic phases that include said additive material,**”

255. Xiao discloses that its invention it adopts a magnesium alloy with high aluminum and zinc content and **further adds elements Fe, Cu, Ni and Ag which can form intermetallics and enhance the corrosion performance of the magnesium alloy.** Xiao explains the corrosion mechanism within a magnesium alloy in terms of a cathode phase, which Xiao explains includes “the  $\beta$  ( $Mg_{17}Al_{12}$ ) phase and eutectic phase on the grain boundary of the magnesium alloy,” and “the magnesium matrix  $\alpha$  phase which functions as an anode phase, so **that the matrix and the grain boundary of the magnesium alloy form a large amount of micro-batteries, which [micro-batteries] greatly accelerate the corrosion decomposition of magnesium alloy.**” *Id.* at ¶ 0026.

256. Xiao discloses that “[e]lements such as **Fe, Cu, Ni, Ag**, etc., in the magnesium alloy **can form a large number of intermetallic composite micro-particles**, which can improve the corrosion performance of the magnesium alloy, thereby promoting the decomposition of the magnesium alloy.” *Id.* at ¶ 0026. A POSITA in August 2014 would have understood this disclosure of “intermetallic composite micro-particles” to be a reference to intermetallic precipitate. As noted above, at least Fe, Cu, and Ni are “additive materials.” Consequently, the in situ precipitation of

Attorney Docket No. 2189.006

Patent 10,329,653

galvanically-active intermetallic phases will include one or more of these additive materials. Thus, Xiao discloses all of the limitations of this claim element.

- h. “said additive material located in sufficient quantities in said galvanically-active intermetallic phases so as to obtain a composition and morphology of said galvanically-active intermetallic phases such that a galvanic corrosion rate along said galvanically-active intermetallic phases causes said magnesium composite to have a dissolution rate of at least at least 5 mg/cm<sup>2</sup>/hr. in 3 wt % KCl water mixture at 90°C.”**

257. As already noted above, other limitations in this claim require that “additive material” be limited to 0.05 wt% of one or more of copper, nickel, zirconium and/or titanium metals. Claim 2 of Xiao discloses: a “magnesium alloy, according to claim 1, further comprising the trace elements at the weight percentages as follows: Fe: 0.1 to 5%, **Cu: 0.05 to 5%, Ni: 0.05 to 5%, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%**; and a sum of the weight percentages of the components is 100%.” *Id.* at Claim 2. Thus, Xiao discloses “sufficient quantities” of each of the copper, nickel, zirconium and titanium (which has been defined by the Patent Owner as “enough additive material” (see Paragraph 78 of my Declaration)).

258. Xiao further discloses that “[e]lements such as Fe, **Cu, Ni**, Ag, etc., in the magnesium alloy **can form a large number of intermetallic composite micro-particles, which can improve the corrosion performance of the magnesium alloy, thereby promoting the decomposition of the magnesium alloy.**” *Id.* ¶ 0026. A POSITA would understand that the galvanically-active intermetallic phases (“composite micro-particles”) have a morphology that enhances the corrosion dissolution rate of the alloy due to a larger number of smaller particles throughout the microstructure. A POSITA would also understand the relationship between “composite micro-particles” and morphology of the galvanically-active intermetallic phases. Thus, Xiao further teaches these limitations of this claim element.

Attorney Docket No. 2189.006

Patent 10,329,653

259. A POSITA in April 2014 would have understood the English translation of Xiao to disclose a table of dissolution rates (“decomposition rates”) because the units of measurement are the units used by POSITAs to measure dissolution rates. In particular, Xiao provides dissolution rates for its various examples, as follows:

	Room temperature tensile strength $\sigma_b$ (MPa)	Decomposition rate at 70 °C in 3% KCl solution (g.cm <sup>-2</sup> .h <sup>-1</sup> )	Decomposition rate at 93 °C in 3% KCl solution (g.cm <sup>-2</sup> .h <sup>-1</sup> )
Comparative example 1	232	0.00026	0.0005
Example 1	360	0.035	0.074
Example 2	385	0.015	0.045
Example 3	410	0.013	0.036
Example 4	375	0.034	0.058
Example 5	392	0.025	0.048
Example 6	365	0.021	0.063
Example 7	387	0.036	0.057

Xiao at ¶ 0064.

260. Xiao provides dissolution rates in g/cm<sup>2</sup>/hr, while the claims of the '653 Patent state dissolution rates in mg/cm<sup>2</sup>/hr. A POSITA in April 2014 would have known that converting from g/cm<sup>2</sup>/hr to mg/cm<sup>2</sup>/hr, merely requires multiplication of the numbers disclosed in this table in Xiao by 1,000.

261. Thus, the dissolution rates disclosed in Xiao convert to:

Attorney Docket No. 2189.006

Patent 10,329,653

	Decomposition rate at 70°C in 3% KCl solution mg/cm <sup>2</sup> /hr	Decomposition rate at 93°C in 3% KCl solution mg/cm <sup>2</sup> /hr
Comparative example 1	0.26	0.5
Example 1	35	74
Example 2	15	45
Example 3	13	36
Example 4	34	58
Example 5	25	48
Example 6	21	63
Example 7	36	57

262. Each of the seven examples (excluding the comparative [prior art] example) provided in Xiao therefore discloses a dissolution rate of “at least 5 mg/cm<sup>2</sup>/hr in 3 wt.% KCl water” at both 70°C or 93°C. Given that the disclosed dissolution rates of Xiao both above and below 90°C are well above the claimed floor of “at least 5 mg/cm<sup>2</sup>/hr” (i.e. at least three times overall and for Example 7 at least seven times) it is my opinion that a POSITA in April 2014 would have recognized the dissolution rate of the examples in Xiao at 93°C to also be necessarily well above the claimed floor of “at least of 5 mg/cm<sup>2</sup>/hr.” Consequently, this element of the claim is also disclosed by Xiao.

263. Accordingly, as set forth in the immediately foregoing paragraphs, it is my opinion that Xiao would have fully disclosed each and every element of Claim 73 of the ‘653 Patent to a POSITA in or before April 2014 in accordance with Patent Owner’s definitions (set forth in Paragraph 78 of my Declaration).

Attorney Docket No. 2189.006

Patent 10,329,653

**B. Ground II: Claims 8, 25, 33, 55, and 74 are anticipated, or at least made obvious, by Xiao.**

264. It is my opinion that Xiao either anticipates or least makes obvious Claims 25, 33, 55, and 74 of the '653 Patent.

- 1. Dependent Claim 8 - “The magnesium composite as defined in claim 1, wherein said magnesium composite is subjected to a surface treatment to improve a surface hardness of said magnesium composite, said surface treatment including peening, heat treatment, aluminizing, or combinations thereof.”**

265. As set forth above, it is my opinion that Xiao would have disclosed all elements of Claim 1 to a POSITA in April 2014.

266. Xiao does not disclose or otherwise suggest subjecting a magnesium composite to a surface treatment (i.e. peening, heat treatment, aluminizing or combinations thereof) nor does it teach that surface treatment would achieve any of the benefits set forth in Claim 8 of the '653 Patent.

267. Xiao discloses that in a particular U.S. Patent a method is disclosed “for preparing a composite material having a decomposable multilayer coated core-shell structure (U.S. Patent No. US2011/0132143A1, 2011). As discussed within Xiao, this U.S. patent publication discloses an electroless plating process applied on the surface of particles of nanoscale core metal powders (such as magnesium, aluminum, zinc, manganese and alloys thereof) to coat multiple layers of a nano-shell of different metals or metal oxides, such as Al, Ni, Al<sub>2</sub>O<sub>3</sub>, and the like, and then the composite powder is sintered to form a nanocomposite material having certain decomposition feature [sic].” Xiao at ¶ 0002.

268. A POSITA in April 2014 would have understood that the sintering discussed within Xiao was a well-known type of surface treatment that can be used to improve the surface hardness of the sintered metal. See, e.g., Ex. 1203 (“Powder metal technology is well known to the persons

Attorney Docket No. 2189.006

Patent 10,329,653

skilled in the art and generally comprises the formation of metal powders which are compacted and then subjected to an elevated temperature so as to produce a sintered product.”). Consequently, Xiao teaches a magnesium composite subjected to a heat treating surface treatment to improve surface hardness of the magnesium.

## 2. Independent Claim 25

### a. “A dissolvable magnesium alloy composite”

269. Xiao discloses “a **magnesium alloy**...and further adds elements of **Fe, Cu, Ni, and Ag** which can **enhance the corrosion performance** of the magnesium alloy...” Xiao at ¶ 0026, ll. 1–9.

270. Thus, Xiao’s disclosure of a combination of a magnesium alloy and elements of iron, copper, nickel, and silver thus meets the construction of “a dissolvable magnesium composite.”

### b. “for use in a ball or other tool component in a well drilling or completion operation,”

271. Xiao discloses that the cast magnesium alloy “can be used as a tripping ball material for a multi-stage sliding sleeve staged-fracturing technique.” *Id.* at ¶ 0001. A POSITA in August 2014 would have understood that a tripping ball was a type of tool component in an oil well drilling operation.

### c. “said dissolvable magnesium alloy composite comprising at least 85 wt % magnesium;”

272. Claim 1 of Xiao discloses a “magnesium alloy, comprising the components at the weight percentages as follows: **Al: 13 to 25%, Zn: 2 to 15%, the remainder is Mg, and a sum of the weight percentages of the components is 100%.**” As such, the total range of the non-magnesium elements disclosed in Claim 1 of Xiao is 15% (*i.e.*, 13 + 2) to 40% (*i.e.*, 25 + 15).

Attorney Docket No. 2189.006

Patent 10,329,653

Thus, claim 1 of Xiao discloses a range of magnesium including 85 wt %. This disclosure of Xiao overlaps the claimed range.

273. Xiao also discloses a prior art magnesium alloy represented by Mg-Al-Zn (with AZ91D magnesium alloy being the most widely used) wherein the “main components of this alloy and the respective weight percentages are as follows: **Al 8.3 to 9.7, Zn 0.35 to 1.0, Mn 0.15 to 0.5, and the remainder is magnesium.**” *Id. at ¶ 0003*. Additionally, Xiao discloses a comparative example using AZ91D as a comparative alloy, wherein the composition of the AZ91D comparative alloy is **9 wt % Al, 1 wt % Zn, 0.3 wt % Zr, and 0.1 wt % Mn, with the remainder being magnesium.** *Id. at ¶ 0032*. As such, the total range of the non-magnesium elements disclosed in the comparative or prior art alloys of Xiao is 88.8% to 91.2%. Thus, Xiao discloses a range of magnesium of at least 85 wt %.

274. Further, Xiao discloses that a primary difference between this prior art magnesium alloy and the novel magnesium alloys taught by Xiao’s disclosure lies in the addition of metallic elements like Iron, Copper, and/or Nickel into the alloy. In particular, Xiao discloses that “[e]lements such as Fe, Cu, Ni, Ag, etc., in the magnesium alloy can form a large number of intermetallic composite micro-particles, which can improve the corrosion performance of the magnesium alloy, thereby promoting the decomposition of the magnesium alloy.” *Id. at ¶ 0026, ll. 15–18.* Also comparing the constituents of the comparative example AZ91D magnesium alloy with the other examples in Table 1 of Xiao (Xiao at ¶ 0061), the introduction of Iron, Copper, and/or Nickel (alongside the Aluminum, Zinc, Zirconium, and Manganese already found in the prior art) appears to make the surprisingly large improvement in dissolution rates.

d. **“[said dissolvable magnesium alloy composite comprising . . .] one or more metals selected from the group consisting of 0.5-10**

Attorney Docket No. 2189.006

Patent 10,329,653

**wt % aluminum, 0.05-6 wt % zinc, 0.01-3 wt % zirconium, and 0.15-2 wt % manganese;”**

275. Claim 1 of Xiao discloses a “magnesium alloy, comprising the components at the weight percentages as follows: Al: 13 to 25%, **Zn: 2 to 15%**,” and Claim 2 of Xiao discloses secondary metals of “Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%, **Zr: 0.05 to 0.5%**, Ti: 0.05 to 0.5%; and a sum of the weight percentages of the components is 100%.”

276. Xiao also discloses a prior art magnesium alloy represented by Mg-Al-Zn (with AZ91D magnesium alloy being the most widely used) wherein the “main components of this alloy and the respective weight percentages are as follows: **Al 8.3 to 9.7, Zn 0.35 to 1.0, Mn 0.15 to 0.5**, and the remainder is magnesium.” *Id. at ¶ 0003*. Additionally, Xiao discloses a comparative example using AZ91D as a comparative alloy, wherein the composition of the AZ91D comparative alloy is **9 wt % Al, 1 wt % Zn, 0.3 wt % Zr, and 0.1 wt % Mn**, with the remainder being magnesium. *Id. at ¶ 0032*.

277. Further, Xiao discloses that a primary difference between this prior art magnesium alloy and the novel magnesium alloys taught by Xiao’s disclosure lies in the addition of metallic elements like Iron, Copper, and/or Nickel into the alloy. In particular, Xiao discloses that “[e]lements such as Fe, Cu, Ni, Ag, etc., in the magnesium alloy can form a large number of intermetallic composite micro-particles, which can improve the corrosion performance of the magnesium alloy, thereby promoting the decomposition of the magnesium alloy.” *Id. at ¶ 0026, ll. 15–18*. Also comparing the constituents of the comparative example AZ91D magnesium alloy with the other examples in Table 1 of Xiao (Xiao at ¶ 0061), the introduction of Iron, Copper, and/or Nickel (alongside the Aluminum, Zinc, Zirconium, and Manganese already found in the prior art) appears to make the surprisingly large improvement in dissolution rates disclosed by Xiao.

Attorney Docket No. 2189.006

Patent 10,329,653

278. Thus, Xiao discloses a novel magnesium alloy including at least two metals, *i.e.*, zinc and zirconium, at weight percentages within the respective claimed ranges of this claim element of the ‘653 Patent. Xiao also discloses two examples of a prior art magnesium alloy, AZ91D that includes at least four metals, *i.e.*, aluminum, zinc, zirconium, and manganese within the respectively claimed weight percentage ranges of this claim element of the ‘653 Patent. Accordingly, Xiao meets this element of the claim.

- e. “**and [said dissolvable magnesium alloy composite comprising . . .] about 0.05-45 wt % of a secondary metal [ . . . said secondary metal including one or more metals selected from the group consisting of copper, nickel, cobalt, titanium and iron]**”

279. Claim 2 of Xiao discloses a “magnesium alloy according to Claim 1, further comprising the trace elements at the weight percentages as follows: **Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%,** Zr: 0.05 to 0.5%, **Ti: 0.05 to 0.5%**; and a sum of the weight percentages of the components is 100%.” Thus, Xiao teaches four candidates to serve as the secondary metal within the claimed weight percentage [and specific metal] limitations of this claim: Copper (Cu), Nickel (Ni), Titanium (Ti), and Iron (Fe). Moreover, as this claim internally defines “a secondary metal” as “including one or more metals,” Xiao also discloses a range of secondary metal running **from 0.05 wt %** (*i.e.*, the low end of the Copper, Nickel or Titanium ranges) **to 15.5 wt %** (*i.e.*, the accumulation of the high end of the Copper, Nickel, Titanium, and Iron ranges (5 wt % + 5 wt % + 0.5 wt% + 5 wt%)).

- f. “**to form a galvanically-active intermetallic particle that promotes corrosion of said dissolvable magnesium alloy composite,**”

280. Xiao discloses that “[e]lements such as Fe, Cu, Ni, Ag, etc., in the magnesium alloy can form a large number of intermetallic composite micro-particles, which can improve the corrosion performance of the magnesium alloy, thereby promoting the decomposition of the

Attorney Docket No. 2189.006

Patent 10,329,653

**magnesium alloy.”** *Id.* at ¶ 0026. These elements are among the claimed “secondary metals” disclosed by Xiao. Thus, Xiao discloses all of the limitations of this claim element.

- g. **“said secondary metal including one or more metals selected from the group consisting of copper, nickel, cobalt, titanium and iron,”**

281. Claim 2 of Xiao discloses a “magnesium alloy according to Claim 1, further comprising the trace elements at the weight percentages as follows: **Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%**, Zr: 0.05 to 0.5%, **Ti: 0.05 to 0.5%**; and a sum of the weight percentages of the components is 100%.” Thus, Xiao teaches four candidates to serve as the secondary metal of this claim: Copper (Cu), Nickel (Ni), Titanium (Ti), and Iron (Fe). Accordingly, Xiao teaches this element of the claim.

- h. **“said magnesium alloy composite has a dissolution rate of at least 5 mg/cm<sup>2</sup>/hr. in 3 wt % KCl water mixture at 90°C.”**

282. A POSITA in April 2014 would have understood the English translation of Xiao to disclose a table of dissolution rates (“decomposition rates”) because the units of measurement are the units used by POSITAs to measure dissolution rates. In particular, Xiao provides dissolution rates for its various examples, as follows:

Attorney Docket No. 2189.006

Patent 10,329,653

	Room temperature tensile strength $\sigma_b$ (MPa)	Decomposition rate at 70 °C in 3% KCl solution (g.cm <sup>-2</sup> .h <sup>-1</sup> )	Decomposition rate at 93 °C in 3% KCl solution (g.cm <sup>-2</sup> .h <sup>-1</sup> )
Comparative example 1	232	0.00026	0.0005
Example 1	360	0.035	0.074
Example 2	385	0.015	0.045
Example 3	410	0.013	0.036
Example 4	375	0.034	0.058
Example 5	392	0.025	0.048
Example 6	365	0.021	0.063
Example 7	387	0.036	0.057

Xiao at ¶ 0064.

283. Xiao provides dissolution rates in g/cm<sup>2</sup>/hr, while the claims of the '653 Patent express dissolution rates in mg/cm<sup>2</sup>/hr. A POSITA in April 2014 would have known that converting from g/cm<sup>2</sup>/hr to mg/cm<sup>2</sup>/hr, merely requires multiplication of the numbers disclosed in this table in Xiao by 1,000.

284. Thus, the dissolution rates disclosed in Xiao convert to:

	Decomposition rate at 70°C in 3% KCl solution mg/cm <sup>2</sup> /hr	Decomposition rate at 93°C in 3% KCl solution mg/cm <sup>2</sup> /hr
Comparative example 1	0.26	0.5
Example 1	35	74
Example 2	15	45

Attorney Docket No. 2189.006

Patent 10,329,653

Example 3	13	36
Example 4	34	58
Example 5	25	48
Example 6	21	63
Example 7	36	57

285. Each of the seven examples (excluding the comparative [prior art] example) provided in Xiao therefore discloses a dissolution rate of “at least 5 mg/cm<sup>2</sup>/hr in 3 wt.% KCl water” at both 70°C or 93°C. Given that the dissolution rate both above and below 90°C are both well above (at least three times) the claimed floor of “at least 5 mg/cm<sup>2</sup>/hr,” it is my opinion that a POSITA in April 2014 would have expected the dissolution rate of the examples in Xiao at 93°C to also be necessarily well above the claimed floor of “at least of 5 mg/cm<sup>2</sup>/hr.” Consequently, this element of the claim is also disclosed by Xiao.

286. While none of the examples disclosed in Xiao include 85% of magnesium, Xiao discloses that a primary difference between this prior art magnesium alloy and the novel magnesium alloys taught by Xiao’s disclosure lies in the addition of metallic elements like Iron, Copper, and/or Nickel into the alloy which appear to make the surprisingly large improvement in dissolution rates. The improvement is illustrated, for instance, by Example 1 of Xiao which comprises a magnesium alloy composite comprising about 77% magnesium, zinc within the claimed range (0.05-6 wt%), zirconium within the claimed range (0.01-3 wt%), and nickel (“the secondary metal”) within the claimed range (0.05 - 45 wt%). Comparing the constituents of the “comparative example” (which is AZ91D magnesium alloy) with Example 1 (and the other examples) of Table 1 of Xiao (Xiao at ¶ 0061), the introduction of Iron, Copper, and/or Nickel (alongside the Aluminum, Zinc, Zirconium, and Manganese already found in the prior art of the

Attorney Docket No. 2189.006

Patent 10,329,653

“comparative example”) provides for Xiao’s surprising increase in dissolution rate over the prior art. It is my opinion that a POSITA in April 2014 would have recognized that the addition of 8 wt% magnesium (to make the total magnesium content at least 85%) in Example 1 by reducing the weight percentages of Aluminum, Zinc, Silver, Titanium, and Zirconium would result in a composition with a dissolution rate at 90°C in a 3% KCl solution of “at least of 5 mg/cm<sup>2</sup>/hr.”

### 3. Independent Claim 33

#### a. “A dissolvable magnesium alloy composite”

287. Xiao discloses “a magnesium alloy...and further adds elements of **Fe, Cu, Ni, and Ag** which can **enhance the corrosion performance** of the magnesium alloy...” Xiao at ¶ 0026, ll. 1–9.

288. Thus, Xiao’s disclosure of a combination of a magnesium alloy and elements of iron, copper, nickel, and silver thus meets the construction of “a magnesium composite.”

#### b. “for use in a ball or other tool component in a well drilling or completion operation,”

289. Xiao discloses that the cast magnesium alloy, “can be used as a tripping ball material for a multi-stage sliding sleeve staged-fracturing technique.” *Id.* at ¶ 0001. A POSITA would have understood that a tripping ball was a type of tool component in an oil well drilling operation. Thus, Xiao teaches this limitation of the claim.

#### c. “said dissolvable magnesium alloy composite comprising **60-95 wt % magnesium;**”

290. Claim 1 of Xiao discloses a “magnesium alloy, comprising the components at the weight percentages as follows: **Al: 13 to 25%, Zn: 2 to 15%, the remainder is Mg, and a sum of the weight percentages of the components is 100%.**” As such, the total range of the non-magnesium elements disclosed in Claim 1 of Xiao is 15% (*i.e.*, 13 + 2) to 40% (*i.e.*, 25 + 15),

Attorney Docket No. 2189.006

Patent 10,329,653

leaving **85 to 60 wt%** for the **magnesium**. The Examples of Xiao disclose a range of magnesium **from 54.5 wt% - 79.2 wt%**. Thus, Xiao discloses a range of magnesium of 54.5 wt% to 85 wt %, which overlaps and covers most of the claimed range. Accordingly, Xiao teaches this limitation of the claim.

- d. **[said dissolvable magnesium alloy composite comprising . . .] 0.5-10 wt % aluminum; 0.05-6 wt % zinc; 0.15-2 wt % manganese;”**

291. Claim 1 of Xiao discloses a “magnesium alloy, comprising the components at the weight percentages as follows: **Al: 13 to 25%, Zn: 2 to 15%**, the remainder is Mg, and a sum of the weight percentages of the components is 100%.” Thus, Xiao discloses a range of zinc (2-15%) that overlaps and covers most of the claimed range (0.05-6 wt%). Accordingly, Xiao teaches this limitation of the claim.

292. Xiao also discloses a prior art magnesium alloy represented by Mg-Al-Zn (with AZ91D magnesium alloy being the most widely used) wherein the “main components of this alloy and the respective weight percentages are as follows: **Al 8.3 to 9.7, Zn 0.35 to 1.0, Mn 0.15 to 0.5**, and the remainder is magnesium.” *Id. at ¶ 0003*. Thus, Xiao also discloses the inclusion of 8.3 to 9.7 wt % aluminum, 0.35 to 1.0 wt % zinc, and 0.15 to 0.5 wt% manganese within each of the specifically claimed ranges in this claim element of the ‘653 Patent.

293. Xiao discloses that a primary difference between this prior art magnesium alloy and the novel magnesium alloys taught by Xiao’s disclosure lies in the addition of metallic elements like Iron, Copper, and/or Nickel into the alloy, which “improve the corrosion performance of the magnesium alloy, thereby promoting the decomposition of the magnesium alloy.” *Id. at ¶ 0026, ll. 15–18*. Also comparing the constituents of “Comparative example 1” in Xiao (an AZ91D magnesium alloy) with the other examples in Table 1 of Xiao (Xiao at ¶ 0061) the introduction of

Attorney Docket No. 2189.006

Patent 10,329,653

Iron, Copper, and/or Nickel (alongside the Aluminum, Zinc and Manganese already found in the prior art) appears to make the surprisingly large improvement in dissolution rates. A POSITA in April 2014 would have recognized that the advantages of the novel magnesium composite found in the addition of iron copper, and/or nickel would have also directly benefited the prior art example and, thus, a POSITA would have recognized the desirability of combining those separate disclosures in Xiao.

294. Thus, Xiao discloses a dissolvable magnesium alloy composite including weight ranges of aluminum, zinc and manganese, which overlap at least a portion of the ranges required by this claim element for those metallic elements. Accordingly, Xiao teaches all of these limitations of the claim element.

- e. “and [said dissolvable magnesium alloy composite comprising . . .] **about 0.05-45 wt % of a secondary metal** [. . . said secondary metal including one or more metals selected from the group consisting of copper, nickel, cobalt, titanium and iron]”

295. Claim 2 of Xiao discloses a “magnesium alloy according to claim 1 further comprising trace elements at the weight percentages as follows: **Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%**, Zr: 0.05 to 0.5%, **Ti: 0.05 to 0.5%**; and a sum of the weight percentages of the components is 100%.” Thus, Xiao teaches four candidates to serve as the secondary metal within the claimed weight percentage [and specific metal] limitations of this claim: **Copper (Cu), Nickel (Ni), Titanium (Ti), and Iron (Fe)**. Moreover, as this claim internally defines “a secondary metal” as “including one or more metals,” Xiao also discloses a range of secondary metal running **from 0.05 wt %** (*i.e.*, the low end of the Copper, Nickel or Titanium ranges) **to 15.5 wt %** (*i.e.*, the accumulation of the high end of the Copper, Nickel, Titanium, and Iron ranges (5 wt % + 5 wt % + 0.5 wt% + 5 wt%)). Hence, Xiao discloses the inclusion of secondary metal in a magnesium composite (including one or more metals (*i.e.*, copper, nickel, titanium, and iron)) in amounts that

Attorney Docket No. 2189.006

Patent 10,329,653

overlap at least a portion of the range required by this claim element. Accordingly, Xiao teaches this limitation of the claim.

- f. “[a secondary metal] to form a galvanically-active intermetallic particle that promotes corrosion of said dissolvable magnesium alloy composite,”

296. Xiao discloses that its invention it adopts a magnesium alloy with high aluminum and zinc content and further adds elements Fe, Cu, Ni and Ag which can form intermetallics and enhance the corrosion performance of the magnesium alloy. Xiao explains the corrosion mechanism within a magnesium alloy in terms of a cathode phase, which Xiao explains includes “the  $\beta$  ( $Mg_{17}Al_{12}$ ) phase and eutectic phase on the grain boundary of the magnesium alloy,” and “the magnesium matrix  $\alpha$  phase which functions as an anode phase, so that the matrix and the grain boundary of the magnesium alloy form a large amount of micro-batteries, which [micro-batteries] greatly accelerate the corrosion decomposition of magnesium alloy.” *Id.* at ¶ 0026.

297. Xiao discloses that “[e]lements such as Fe, Cu, Ni, Ag, etc., in the magnesium alloy can form a large number of intermetallic composite micro-particles, which can improve the corrosion performance of the magnesium alloy, thereby promoting the decomposition of the magnesium alloy.” *Id.* at ¶ 0026. These elements are among the claimed “secondary metals” disclosed by Xiao. Thus, all of the limitations of this claim element are disclosed by Xiao.

- g. “said secondary metal including one or more metals selected from the group consisting of copper, nickel, cobalt, titanium and iron,”

298. Xiao discloses one or more of the “secondary metals,” including copper (Cu), nickel (Ni), titanium (Ti) and iron (Fe) as follows: “**Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%**, Zr: 0.05 to 0.5%, **Ti: 0.05 to 0.5%**.” Xiao at Claim 2. Of the additive materials taught in

Attorney Docket No. 2189.006

Patent 10,329,653

Example 7 of Xiao (i.e. Al-Fe, Al-Ni, Al-Ag, Al-Cu, Al-Zr, Al-Ti and Zinc), **Al-Fe**, **Al-Ni**, **Al-Cu**, and **Al-Ti** contain **copper, nickel, titanium and iron**. This claim element only requires the secondary metal include one of these metals. Thus, Xiao teaches four candidates to serve as the secondary metal of this claim: Copper (Cu), Nickel (Ni), Titanium (Ti), and Iron (Fe).

**h. “said magnesium alloy composite has a dissolution rate of at least 5 mg/cm<sup>2</sup>/hr. in 3 wt % KCl water mixture at 90°C.”**

299. A POSITA in April 2014 would have understood the English translation of Xiao to disclose a table of dissolution rates (“decomposition rates”) because the units of measurement are the units used by POSITAs to measure dissolution rates. In particular, Xiao provides dissolution rates for its various examples, as follows:

	Room temperature tensile strength $\sigma_b$ (MPa)	Decomposition rate at 70 °C in 3% KCl solution (g.cm <sup>-2</sup> .h <sup>-1</sup> )	Decomposition rate at 93 °C in 3% KCl solution (g.cm <sup>-2</sup> .h <sup>-1</sup> )
Comparative example 1	232	0.00026	0.0005
Example 1	360	0.035	0.074
Example 2	385	0.015	0.045
Example 3	410	0.013	0.036
Example 4	375	0.034	0.058
Example 5	392	0.025	0.048
Example 6	365	0.021	0.063
Example 7	387	0.036	0.057

Xiao at ¶ 0064.

Attorney Docket No. 2189.006

Patent 10,329,653

300. Xiao provides dissolution rates in g/cm<sup>2</sup>/hr, while the claims of the '653 Patent express dissolution rates in mg/cm<sup>2</sup>/hr. A POSITA in April 2014 would have known that converting from g/cm<sup>2</sup>/hr to mg/cm<sup>2</sup>/hr, merely requires multiplication of the numbers disclosed in this table in Xiao by 1,000.

301. Thus, the dissolution rates disclosed in Xiao convert to:

	Decomposition rate at 70°C in 3% KCl solution mg/cm <sup>2</sup> /hr	Decomposition rate at 93°C in 3% KCl solution mg/cm <sup>2</sup> /hr
Comparative example 1	0.26	0.5
Example 1	35	74
Example 2	15	45
Example 3	13	36
Example 4	34	58
Example 5	25	48
Example 6	21	63
Example 7	36	57

302. Each of the seven examples (excluding the comparative [prior art] example) provided in Xiao therefore discloses a dissolution rate of "at least 5 mg/cm<sup>2</sup>/hr in 3 wt.% KCl water" at both 70°C or 93°C. Given that the disclosed dissolution rates of Xiao both above and below 90°C are well above the claimed floor of "at least 5 mg/cm<sup>2</sup>/hr" (i.e. at least three times overall and for Example 7 at least seven times) it is my opinion that a POSITA in April 2014 would have recognized the dissolution rate of the examples in Xiao at 93°C to also be necessarily well above the claimed floor of "at least of 5 mg/cm<sup>2</sup>/hr." Consequently, this element of Claim 1 is also disclosed by Xiao.

Attorney Docket No. 2189.006

Patent 10,329,653

303. Accordingly, as set forth in the immediately foregoing paragraphs, it is my opinion that Xiao would have fully disclosed each and every element of Claim 33 of the ‘653 Patent to a POSITA in or before April 2014 in accordance with Patent Owner’s definitions (set forth in Paragraph 78 of my Declaration).

#### 4. Dependent Claim 55

- a. “The magnesium composite as defined in claim 49, wherein said magnesium alloy includes at least 85 wt. % magnesium,”

304. As set forth above, it is my opinion that Xiao would have disclosed all elements of Claim 49 to a POSITA in April 2014.

305. Claim 1 of Xiao discloses a “magnesium alloy, comprising the components at the weight percentages as follows: **Al: 13 to 25%, Zn: 2 to 15%, the remainder is Mg, and a sum of the weight percentages of the components is 100%.**” As such, the total range of the non-magnesium elements disclosed in Claim 1 of Xiao is 15% (*i.e.*, 13 + 2) to 40% (*i.e.*, 25 + 15). Thus, claim 1 of Xiao discloses a range of magnesium including 85 wt %. This disclosure of Xiao overlaps the claimed range.

306. Xiao also discloses a prior art magnesium alloy represented by Mg-Al-Zn (with AZ91D magnesium alloy being the most widely used) wherein the “main components of this alloy and the respective weight percentages are as follows: **Al 8.3 to 9.7, Zn 0.35 to 1.0, Mn 0.15 to 0.5, and the remainder is magnesium.**” *Id.* at ¶ 0003. Additionally, Xiao discloses a comparative example using AZ91D as a comparative alloy, wherein the composition of the AZ91D comparative alloy is **9 wt % Al, 1 wt % Zn, 0.3 wt % Zr, and 0.1 wt % Mn, with the remainder being magnesium.** *Id.* at ¶ 0032. As such, the total range of the non-magnesium elements disclosed in the comparative or prior art alloys of Xiao is 88.8% to 91.2%. Thus, Xiao discloses a range of magnesium of at least 85 wt %.

Attorney Docket No. 2189.006

Patent 10,329,653

307. Further, Xiao discloses that a primary difference between this prior art magnesium alloy and the novel magnesium alloys taught by Xiao's disclosure lies in the addition of metallic elements like Iron, Copper, and/or Nickel into the alloy. In particular, Xiao discloses that “[e]lements such as Fe, Cu, Ni, Ag, etc., in the magnesium alloy can form a large number of intermetallic composite micro-particles, which can improve the corrosion performance of the magnesium alloy, thereby promoting the decomposition of the magnesium alloy.” *Id.* at ¶ 0026, ll. 15–18. Also comparing the constituents of the comparative example AZ91D magnesium alloy with the other examples in Table 1 of Xiao (Xiao at ¶ 0061), the introduction of Iron, Copper, and/or Nickel (alongside the Aluminum, Zinc, Zirconium, and Manganese already found in the prior art) appears to make the surprisingly large improvement in dissolution rates.

**b. “and one or more metals selected from the group consisting of 0.5-10 wt. % aluminum, 0.05-6 wt. % zinc, 0.01-3 wt. % zirconium, and 0.15-2 wt. % manganese.”**

308. Claim 1 of Xiao discloses a “magnesium alloy, comprising the components at the weight percentages as follows: Al: 13 to 25%, **Zn: 2 to 15%**,” and Claim 2 of Xiao discloses secondary metals of “Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%, **Zr: 0.05 to 0.5%**, Ti: 0.05 to 0.5%; and a sum of the weight percentages of the components is 100%.”

309. Xiao also discloses a prior art magnesium alloy represented by Mg-Al-Zn (with AZ91D magnesium alloy being the most widely used) wherein the “main components of this alloy and the respective weight percentages are as follows: **Al 8.3 to 9.7, Zn 0.35 to 1.0, Mn 0.15 to 0.5**, and the remainder is magnesium.” *Id.* at ¶ 0003. Additionally, Xiao discloses a comparative example using AZ91D as a comparative alloy, wherein the composition of the AZ91D comparative alloy is **9 wt % Al, 1 wt % Zn, 0.3 wt % Zr, and 0.1 wt % Mn**, with the remainder being magnesium. *Id.* at ¶ 0032.

Attorney Docket No. 2189.006

Patent 10,329,653

310. Further, Xiao discloses that a primary difference between this prior art magnesium alloy and the novel magnesium alloys taught by Xiao's disclosure lies in the addition of metallic elements like Iron, Copper, and/or Nickel into the alloy. In particular, Xiao discloses that “[e]lements such as Fe, Cu, Ni, Ag, etc., in the magnesium alloy can form a large number of intermetallic composite micro-particles, which can improve the corrosion performance of the magnesium alloy, thereby promoting the decomposition of the magnesium alloy.” *Id.* at ¶ 0026, ll. 15–18. Also comparing the constituents of the comparative example AZ91D magnesium alloy with the other examples in Table 1 of Xiao (Xiao at ¶ 0061), the introduction of Iron, Copper, and/or Nickel (alongside the Aluminum, Zinc, Zirconium, and Manganese already found in the prior art) appears to make the surprisingly large improvement in dissolution rates disclosed by Xiao.

311. Thus, Xiao discloses a novel magnesium alloy including at least two metals, *i.e.*, zinc and zirconium, at weight percentages within the respective claimed ranges of this claim element of the ‘653 Patent. Xiao also discloses two examples of a prior art magnesium alloy, AZ91D that includes at least four metals, *i.e.*, aluminum, zinc, zirconium, and manganese within the respectively claimed weight percentage ranges of this claim element of the ‘653 Patent. Accordingly, Xiao discloses all of the limitations of this claim element.

## 5. Independent Claim 74

### a. “A dissolvable magnesium composite”

312. Xiao discloses “a **magnesium alloy**...and further adds elements of **Fe, Cu, Ni, and Ag** which can **enhance the corrosion performance** of the magnesium alloy...” Xiao at ¶ 0026, ll. 1–9.

Attorney Docket No. 2189.006

Patent 10,329,653

313. Thus, Xiao's disclosure of a combination of a magnesium alloy and elements of iron, copper, nickel, and silver thus meets the construction of "a dissolvable magnesium composite."

- b. **"for use in a ball or other tool component in a well drilling or completion operation,"**

314. Xiao discloses a cast magnesium alloy that "**can be used as a tripping ball material for a multi-stage sliding sleeve staged-fracturing technique.**" *Id.* at ¶ 0001. A POSITA would have understood that a tripping ball was a type of tool component in an oil well drilling operation. Thus, Xiao teaches this limitation of the claim.

- c. **"said dissolvable magnesium composite includes in situ precipitation of galvanically-active intermetallic phases to enable controlled dissolution of said magnesium composite,"**

315. Xiao discloses that its invention it adopts a magnesium alloy with high aluminum and zinc content and **further adds elements Fe, Cu, Ni and Ag which can form intermetallics and enhance the corrosion performance of the magnesium alloy.** Xiao explains the corrosion mechanism within a magnesium alloy in terms of a cathode phase, which Xiao explains includes "the  $\beta$  ( $Mg_{17}Al_{12}$ ) phase and eutectic phase on the grain boundary of the magnesium alloy," and "the magnesium matrix  $\alpha$  phase which functions as an anode phase, so **that the matrix and the grain boundary of the magnesium alloy form a large amount of micro-batteries, which [micro-batteries] greatly accelerate the corrosion decomposition of magnesium alloy.**" *Id.* at ¶ 0026.

316. Xiao discloses that "[e]lements such as Fe, Cu, Ni, Ag, etc., in the magnesium alloy **can form a large number of intermetallic composite micro-particles, which can improve the corrosion performance of the magnesium alloy, thereby promoting the decomposition of the magnesium alloy.**" *Id.* at ¶ 0026, ll. 15–18. A POSITA in August 2014 would have understood

Attorney Docket No. 2189.006

Patent 10,329,653

this disclosure of “intermetallic composite micro-particles” to be a reference to intermetallic precipitate.

317. A POSITA reading the Xiao reference in April 2014 would have recognized that the precipitation of galvanically-active intermetallic phases (“micro-batteries”) are necessarily generated by the *in situ* reaction between magnesium and any and all of the disclosed metal additives forming *in situ* precipitate. Thus, all of the limitations of this claim element are disclosed by Xiao.

- d. “said magnesium composite comprising a mixture of magnesium or a **magnesium alloy** and an **additive material**,”

318. Xiao discloses “first loading **pure magnesium and pure aluminum** into a smelting furnace . . . , **next loading** pure zinc and **an [sic] intermediate alloys** of trace element components into a **resulting magnesium-aluminum alloy melt** after melting.” *Id.* at ¶ 0022.

319. Xiao discloses that these “intermediate alloys” are **Al-Fe, Al-Ni, Al-Cu, Al-Ag, Al-Zr, Al-Ti**, which are heated to dry and then added to the magnesium-aluminum alloy melt.” *Id.* at ¶ 0023. These intermediate alloys meet Patent Owner’s proposed definition of “additive material,” i.e. “a material that is added.”

320. The addition of one or more of these intermediate alloys to the melted magnesium alloy necessarily results in a mixture of magnesium or magnesium alloy and the additive material. Thus, Xiao discloses all of the limitations of this claim element.

- e. “said **additive material** constituting **at least 0.1 wt %** of said mixture, [...said additive material is a metal material selected from the group consisting of copper, nickel, and cobalt]”

321. Claim 1 of Xiao discloses: a “magnesium alloy, comprising the components at the weight percentages as follows: Al: **13 to 25%**, Zn: **2 to 15%**, the remainder is Mg, and a sum of the weight percentages of the components is 100%.” Xiao at Claim 1. And Claim 2 discloses: a

Attorney Docket No. 2189.006

Patent 10,329,653

“magnesium alloy, according to claim 1, further comprising the trace elements at the weight percentages as follows: Fe: 0.1 to 5%, **Cu: 0.05 to 5%, Ni: 0.05 to 5%**, Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%; and a sum of the weight percentages of the components is 100%.” *Id.* at Claim 2. Thus, Xiao discloses two metals that could serve as the claimed “additive material” and have weight % quantities that fall within the range of this claim element.

322. These metallic elements are found in the additive materials generally disclosed by Xiao (*i.e.*, **intermediate alloys: Al-Fe, Al-Ni, Al-Cu, Al-Ti**) (Xiao at ¶ 0023) and particularly described in association with Example 7 of Xiao (*Id.* at ¶¶ 0057 - 0060) for which I have already calculated (and set forth above) the specific weight percentage (ranges) of each additive material (which has been defined by the Patent Owner as “a material that is added” (see Paragraph 78 of my Declaration)) set forth in Example 7 of Xiao (*i.e.* Al-Fe, Al-Ni, Al-Ag, Al-Cu, Al-Zr, Al-Ti and Zinc).

323. In particular, the additive materials aluminum-iron with 30 wt% iron, aluminum-nickel with 45 wt% nickel, and aluminum-copper with 40 wt% copper, which each have greater melting point temperatures than the solidus temperature of magnesium or the magnesium aluminum alloy disclosed in Xiao, also fall well within the range of about 0.05 wt% to 45 wt% of the mixture. For example, the aluminum-iron additive constitutes **3.3 wt% of the mixture** (*i.e.*, 1%/0.30); the aluminum-nickel additive constitutes **1.1 wt% of the mixture** (*i.e.*, 0.5%/0.45); and the aluminum-copper additive constitutes **0.25 wt% of the mixture** (*i.e.*, 0.1%/0.40). A POSITA in April 2014 would have also recognized that other intermediate alloy compositions would have fallen within the teachings of Example 7 of Xiao as well as the other teachings of Xiao and resulted in **Al-Fe, Al-Ni, and Al-Cu intermediate alloys** constituting **at least 0.1 wt%** of the mixture. Accordingly, this element of the claim is disclosed by Xiao. These metallic elements are found in

Attorney Docket No. 2189.006

Patent 10,329,653

the intermediate alloys: Al-Fe, Al-Ni, Al-Cu, Al-Ag, Al-Zr, Al-Ti (i.e., additives) of Xiao. Xiao at ¶ 0023.

**f. “said magnesium in said magnesium composite constituting at least 85 wt %;”**

324. Claim 1 of Xiao discloses a “magnesium alloy, comprising the components at the weight percentages as follows: **Al: 13 to 25%, Zn: 2 to 15%, the remainder is Mg, and a sum of the weight percentages of the components is 100%.**” As such, the total range of the non-magnesium elements disclosed in Claim 1 of Xiao is 15% (i.e., 13 + 2) to 40% (i.e., 25 + 15). Thus, claim 1 of Xiao discloses a range of magnesium including 85 wt %. This disclosure of Xiao overlaps the claimed range.

325. Xiao also discloses a prior art magnesium alloy represented by Mg-Al-Zn (with AZ91D magnesium alloy being the most widely used) wherein the “main components of this alloy and the respective weight percentages are as follows: **Al 8.3 to 9.7, Zn 0.35 to 1.0, Mn 0.15 to 0.5, and the remainder is magnesium.**” *Id. at ¶ 0003.* Additionally, Xiao discloses a comparative example using AZ91D as a comparative alloy, wherein the composition of the AZ91D comparative alloy is **9 wt % Al, 1 wt % Zn, 0.3 wt % Zr, and 0.1 wt % Mn, with the remainder being magnesium.** *Id. at ¶ 0032.* As such, the total range of the non-magnesium elements disclosed in the comparative or prior art alloys of Xiao is 88.8% to 91.2%. Thus, Xiao discloses a range of magnesium of at least 85 wt %.

326. Further, Xiao discloses that a primary difference between this prior art magnesium alloy and the novel magnesium alloys taught by Xiao’s disclosure lies in the addition of metallic elements like Iron, Copper, and/or Nickel into the alloy. In particular, Xiao discloses that “[e]lements such as Fe, Cu, Ni, Ag, etc., in the magnesium alloy can form a large number of intermetallic composite micro-particles, which can improve the corrosion performance of the

Attorney Docket No. 2189.006

Patent 10,329,653

magnesium alloy, thereby promoting the decomposition of the magnesium alloy.” *Id.* at ¶ 0026, ll. 15–18. Also comparing the constituents of the comparative example AZ91D magnesium alloy with the other examples in Table 1 of Xiao (Xiao at ¶ 0061), the introduction of Iron, Copper, and/or Nickel (alongside the Aluminum, Zinc, Zirconium, and Manganese already found in the prior art) appears to make the surprisingly large improvement in dissolution rates.

- g. “said **additive material** is a metal material selected from the group consisting of **copper, nickel, and cobalt,**”

327. Claim 2 of Xiao discloses a “magnesium alloy according to Claim 1, further comprising the trace elements at the weight percentages as follows: Fe: 0.1 to 5%, **Cu: 0.05 to 5%, Ni: 0.05 to 5%,** Zr: 0.05 to 0.5%, Ti: 0.05 to 0.5%; and a sum of the weight percentages of the components is 100%.” This claim element requires that the additive material be one of the metals in the claimed group. Thus, Xiao teaches two candidates to serve as the additive material meeting this limitation secondary metal of the claim: Copper (Cu) and Nickel (Ni).

- h. “said magnesium composite including in situ precipitation of galvanically-active intermetallic phases that include said **additive material,**”

328. Xiao discloses that its invention it adopts a magnesium alloy with high aluminum and zinc content and **further adds elements Fe, Cu, Ni and Ag which can form intermetallics** and **enhance the corrosion performance of the magnesium alloy.** Xiao explains the corrosion mechanism within a magnesium alloy in terms of a cathode phase, which Xiao explains includes “the  $\beta$  ( $Mg_{17}Al_{12}$ ) phase and eutectic phase on the grain boundary of the magnesium alloy,” and “the magnesium matrix  $\alpha$  phase which functions as an anode phase, so **that the matrix and the grain boundary of the magnesium alloy form a large amount of micro-batteries, which [micro-batteries] greatly accelerate the corrosion decomposition of magnesium alloy.**” *Id.* at ¶ 0026.

Attorney Docket No. 2189.006

Patent 10,329,653

329. Xiao discloses that “[e]lements such as Fe, **Cu**, **Ni**, Ag, etc., in the magnesium alloy **can form a large number of intermetallic composite micro-particles**, which can improve the corrosion performance of the magnesium alloy, thereby promoting the decomposition of the magnesium alloy.” *Id.* at ¶ 0026.

330. A POSITA reading the Xiao reference in April 2014 would have recognized that the precipitation of galvanically-active intermetallic phases (“micro-batteries”) are necessarily generated by the *in situ* reaction between magnesium Mg and each of the disclosed metal additives forming *in situ* precipitate that includes the additive material (*i.e.*, Cu or Ni) in place. Thus, this claim element was fully disclosed by Xiao.

- i. **“said magnesium composite has a dissolution rate of 84-325 mg/cm<sup>2</sup>/hr. in 3 wt % KCl water mixture at 90°C.”**

331. A POSITA in April 2014 would have understood the English translation of Xiao to disclose a table of dissolution rates (“decomposition rates”) because the units of measurement in that table are units regularly used by POSITAs to express measurements of dissolution. In particular, Xiao provides dissolution rates for its various examples, as follows:

Attorney Docket No. 2189.006

Patent 10,329,653

	Room temperature tensile strength $\sigma_b$ (MPa)	Decomposition rate at 70 °C in 3% KCl solution (g.cm <sup>-2</sup> .h <sup>-1</sup> )	Decomposition rate at 93 °C in 3% KCl solution (g.cm <sup>-2</sup> .h <sup>-1</sup> )
Comparative example 1	232	0.00026	0.0005
Example 1	360	0.035	0.074
Example 2	385	0.015	0.045
Example 3	410	0.013	0.036
Example 4	375	0.034	0.058
Example 5	392	0.025	0.048
Example 6	365	0.021	0.063
Example 7	387	0.036	0.057

*Id.* at ¶ 0064.

332. Xiao provides dissolution rates in g/cm<sup>2</sup>/hr, while the claims of the '653 patent express the claimed dissolution rate in mg/cm<sup>2</sup>/hr. A POSITA in April 2014 would have known that converting from g/cm<sup>2</sup>/hr to mg/cm<sup>2</sup>/hr, merely requires multiplication of the numbers disclosed in this table from Xiao by 1,000.

333. Thus, the dissolution rates disclosed in Xiao convert to:

	Decomposition rate at 70°C in 3% KCl solution mg/cm <sup>2</sup> /hr	Decomposition rate at 93°C in 3% KCl solution mg/cm <sup>2</sup> /hr
Comparative example 1	0.26	0.5
Example 1	35	74
Example 2	15	45

Attorney Docket No. 2189.006

Patent 10,329,653

Example 3	13	36
Example 4	34	58
Example 5	25	48
Example 6	21	63
Example 7	36	57

334. The examples (excluding the comparative [prior art] example) provided in Xiao therefore expressly disclose dissolution rates up to 74 mg/cm<sup>2</sup>/hr in 3 wt.% KCl water at 93°C.

335. It is my opinion that a POSITA reading the Xiao reference in April 2014 would have understood that Xiao additionally discloses dissolution rates above the claimed floor of 84 mg/cm<sup>2</sup>/hr at 90°C because (1) the dissolution corrosion rates in the Xiao examples of Xiao are very close to the claimed floor of 84 mg/cm<sup>2</sup>/hr at 90°C (for example, Example 1 exhibited a dissolution rate of 74 mg/cm<sup>2</sup>/hr at 93°C, Example 6 a dissolution rate of 63 mg/cm<sup>2</sup>/hr at 93°C, and Example 7 a dissolution rate of 57 mg/cm<sup>2</sup>/hr at 93°C (Xiao at 0026)); and (2) a POSITA in April 2014 would have expected that other magnesium compositions having additive amounts in the ranges as disclosed by Xiao, but with higher amounts of copper, nickel, and/or iron than the amounts used in the tested Xiao examples would improve corrosion performance. In particular, even though they come close to the 84 mg/cm<sup>2</sup>/hr at 90°C, Examples 1, 6 and 7 of the Xiao examples had less aggregate copper, nickel and iron (as shown in the table here:

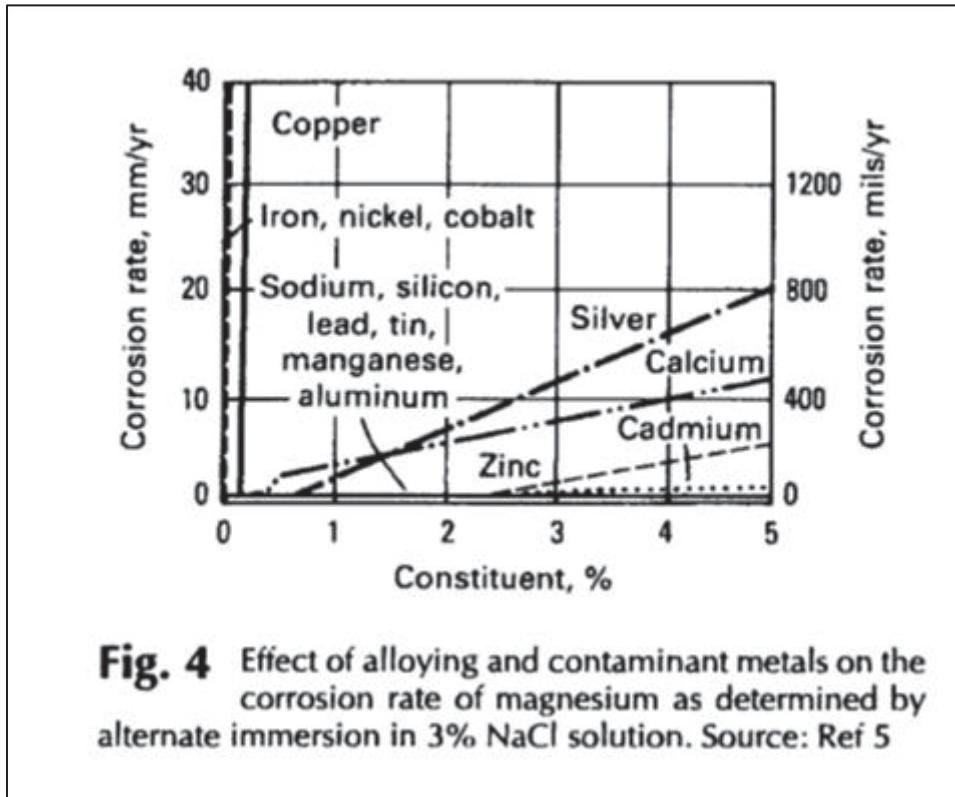
	wt% Copper	wt% Nickel	wt% Iron
<b>Example 1</b>	0	5	0.1
<b>Example 6</b>	1	0.2	1.5
<b>Example 7</b>	0.1	0.5	1

Attorney Docket No. 2189.006

Patent 10,329,653

than taught by Claim 2 of Xiao which says that there can be 5 wt% for each of copper, nickel, and iron in a magnesium composite. Thus, the Examples in Xiao were using lower amounts of these specific dissolution-enhancing additive elements in assessing dissolution rates. It is my opinion that a POSITA in April 2014 would have understood that Xiao inherently discloses magnesium compositions having dissolution rates necessarily well above the claimed floor of at least 84 mg/cm<sup>2</sup>/hr ranges because Xiao discloses that the dissolution-enhancing additive elements may be present in higher amounts (e.g., up to 5 wt% for **each** of copper, nickel, and iron (*Id.* at Claim 2)), as further evidenced below.

336. Confirming my belief that a dissolution rate of at least 84 mg/cm<sup>2</sup>/hr ranges is inherently disclosed by Xiao, it was well known prior to the priority date of April 2014 that additions of copper, iron, nickel and cobalt cause a sharp linear increase in the corrosion rate of magnesium as evidenced by Shaw. Shaw is a printed publication entitled “Corrosion Resistance of Magnesium Alloys” published in 2003 in the ASM Handbook, Vol. 13A. *Id.* Figure 4 from the Shaw publication, set forth below, illustrates the dramatic and linear effect of small additions of these impurities on magnesium corrosion rates. In addition, the ASM Handbook is a very common reference document used by metallurgical and materials engineers/scientists and would have been well known. As such, the recitation of the claimed dissolution rate of 84-325 mg/cm<sup>2</sup>/hr at 90°C was not a new property or use but rather a property that would be inherent in the compositions disclosed in Xiao, as evidenced by Shaw as published in the ASM Handbook.



Shaw at Figure 4.

**C. Ground III: Claims 7, 12, 13, and 71 are Obvious over Xiao in view of Hassan**

337. It is my opinion that the disclosure of Xiao [Xiao] in combination with the disclosures of Hassan [Hassan] would have rendered Claims 7 and 8 of the '653 Patent obvious to a POSITA in April 2014.

1. **Dependent Claim 7 -- “The magnesium composite as defined in claim 1, where said magnesium composite is subjected to a deformation processing to reduce grain size of said magnesium composite, increase tensile yield strength of said magnesium composite, increase elongation of said magnesium composite, or combinations thereof.”**

338. As set forth above, it is my opinion that Xiao would have disclosed all elements of Claim 1 to a POSITA in April 2014.

339. Xiao does not disclose or otherwise suggest subjecting a magnesium composite to deformation processing nor does it teach that deformation processing would achieve any of the

Attorney Docket No. 2189.006

Patent 10,329,653

benefits set forth in Claim 7 of the ‘653 Patent. Rather, Xiao discloses dissolvable magnesium composites with accelerated dissolution rates and other improved properties. Hassan (like Xiao) discloses magnesium based materials or alloys and teaches that there are benefits to reinforcing these materials or alloys with other metals or metal alloys to improve their properties.

340. Hassan, on the other hand, does teach deformation processing and its benefits with respect to magnesium-based composites reinforced with nickel made using a cast melt technique. Hassan at p. 2467, cols 1–2.

341. It is my opinion that a POSITA in April 2014 would have been motivated to combine the disclosure of Xiao with the disclosure of deformation processing in Hassan rendering Claim 7 obvious. First, Xiao and Hassan are both directed to improving the properties of magnesium based materials, especially those for use in devices or tools. Hassan, for instance, teaches that:

increasing hostile service conditions that the modern engineering devices have to withstand have led the materials scientists across the globe to create new materials with enhanced properties when compared to the conventional materials. One way to improve the properties of conventional metallic materials is to reinforce them judiciously keeping the end application in mind. Among the reinforced metallic materials, magnesium based composites are becoming the strong candidates for lightweight structural application due to their superior specific mechanical properties.

Hassan at 2467. Hassan also recognizes that type of processing, type, size, and volume fraction of reinforcement are “common factors governing the end properties of a metal matrix composite[].”

*Id.*

342. Xiao similarly teaches in the context of its smelt casted magnesium alloy, the use of reinforcing metals or metal alloys to enhance corrosion performance and/or improve the compressive strength of the formed magnesium material. Xiao at ¶ 0026. In particular, Xiao noted that existing magnesium alloys (such as AZ91D) had low compressive strength and slow

Attorney Docket No. 2189.006

Patent 10,329,653

decomposition rates, making them unsuitable as material for, for example, a tripping ball used in oil well drilling. *Id.* at 0003.

343. As discussed at length above, Xiao responded to those recognized problems in the art by “adjusting the chemical composition and preparation process” to prepare a light and pressure-proof cast magnesium alloy that adds elements which can enhance the corrosion performance of the magnesium alloy. *Id.* at 0004.

344. A POSITA would be motivated to modify the disclosed magnesium alloys and magnesium-alloy-based tripping ball material, taught in Xiao to further increase and/or control the rate of dissolution.

345. More specifically, a POSITA would look to Hassan, which teaches pure magnesium reinforced with nickel particulates that exhibits improved physical and mechanical properties over magnesium alloy AZ91. Hassan at p. 2467, col. 2. Hassan also teaches that careful “selection of reinforcing phase remains one of the most critical factors in developing a composite material with superior properties when compared to its monolithic counterparts.” *Id.* Additionally, Hassan notes that its magnesium composites possess improved thermal expansion and mechanical characteristics (*Id.* at p. 2473, col. 2), which would be beneficial in oil field applications. Accordingly, a POSITA would have recognized the benefits set forth in Hassan applied to the magnesium alloy used in the tools taught in Xiao.

346. Hassan teaches the “synthesis of monolithic and nickel reinforced magnesium composites (Mg/Nip),” including heating the magnesium with reinforcement particulates (placed in multi-layer sandwich form) to 750°C, stirring the mixture, treating with argon gas, and depositing the resulting composite onto a metal substrate. *Id.* at 2468.

Attorney Docket No. 2189.006

Patent 10,329,653

347. Importantly for Claim 7 of the ‘653 Patent, Hassan discloses that the deposited magnesium ingots may then be subjected to secondary processing. For example, Hassan teaches that the nickel-reinforced magnesium ingots are machined and hot extruded to obtain rods of 8 mm diameter. *Id.* Analysis of the composite of Hassan “showed the reduction in the size of elemental nickel particulates in the extruded composites” (see Table 1). In other words, the extrusion in Hassan resulted in a reduction of particulate size of the magnesium composite.

348. A POSITA in April 2014 would have been motivated to treat the magnesium alloys disclosed in Xiao using the secondary processing techniques disclosed by Hassan such that the size of additive particulates and magnesium composite could be controlled and optimized. Xiao already disclosed that controlling the particulate size of its magnesium alloy was desirable; Xiao simply teaches the use of argon treatment and a refining agent to do so (rather than the deformation processing claimed in Claim 7 of the ‘653 Patent). Xiao at ¶¶ 0036 and 0044. Accordingly, a POSITA in April 2014 would have been motivated to combine Xiao with Hassan.

349. Moreover, a POSITA would have had a reasonable expectation of successfully combining the magnesium alloy of Xiao with the secondary processing techniques of Hassan. Those secondary processing techniques of Hassan were conventionally used in metal alloy treatment and such processing techniques have been known for decades to optimize particulate size of metal alloys and improve the properties.

350. A POSITA would therefore have had a reasonable expectation of success in making the alloy of Xiao and treating it with a secondary processing technique such as extrusion as disclosed by Hassan and later claimed by Claim 7 of the ‘653 Patent. Accordingly, Claim 7 should be found obvious over Xiao in view of Hassan.

351.

Attorney Docket No. 2189.006

Patent 10,329,653

**2. Independent Claim 12**

**a. “A magnesium composite”**

352. Xiao discloses a “magnesium alloy...and further adds elements of Fe, Cu, Ni, and Ag which can enhance the corrosion performance of the magnesium alloy...” Xiao at ¶ 0026, ll. 1–9.

353. Thus, Xiao’s disclosure of a combination of a magnesium alloy and elements of iron, copper, nickel, and silver thus meets the construction of “a magnesium composite.”

**b. “[magnesium composite] that includes in situ precipitation of galvanically-active intermetallic phases to enable controlled dissolution of said magnesium composite”**

354. Xiao discloses that its invention it adopts a magnesium alloy with high aluminum and zinc content and **further adds elements Fe, Cu, Ni and Ag which can form intermetallics** and **enhance the corrosion performance of the magnesium alloy**. Xiao explains the corrosion mechanism within a magnesium alloy in terms of a cathode phase, which Xiao explains includes “the  $\beta$  ( $Mg_{17}Al_{12}$ ) phase and eutectic phase on the grain boundary of the magnesium alloy,” and “the magnesium matrix  $\alpha$  phase which functions as an anode phase, so **that the matrix and the grain boundary of the magnesium alloy form a large amount of micro-batteries, which [micro-batteries] greatly accelerate the corrosion decomposition of magnesium alloy.**” *Id.* at ¶ 0026.

355. Xiao further discloses that “[e]lements such as Fe, Cu, Ni, Ag, etc., in the magnesium alloy **can form a large number of intermetallic composite micro-particles**, which can improve the corrosion performance of the magnesium alloy, thereby promoting the decomposition of the magnesium alloy.” *Id.* at ¶ 0026, ll 15-18. A POSITA in August 2014 would

Attorney Docket No. 2189.006

Patent 10,329,653

have understood this disclosure of “intermetallic composite micro-particles” to be a reference to intermetallic precipitate.

356. A POSITA reading the Xiao reference in April 2014 would have recognized that the precipitation of galvanically-active intermetallic phases (“micro-batteries”) are naturally and necessarily generated by the *in situ* reaction between magnesium and any and all of the disclosed metal additives forming *in situ* precipitate. Thus, all of the limitations of this claim element are disclosed by Xiao.

c. **“comprising a mixture of a magnesium or a magnesium alloy and an additive material,”**

357. Xiao discloses “first loading **pure magnesium and pure aluminum** into a smelting furnace . . . , **next loading** pure zinc and **an [sic] intermediate alloys** of trace element components into a **resulting magnesium-aluminum alloy melt** after melting.” *Id.* at ¶ 0022.

358. Xiao discloses that these “intermediate alloys” are **Al-Fe, Al-Ni, Al-Cu, Al-Ag, Al-Zr, Al-Ti**, which are heated to dry and then added to the magnesium-aluminum alloy melt.” *Id.* at ¶ 0023. These intermediate alloys meet Patent Owner’s proposed definition of “additive material,” i.e. “a material that is added.”

359. The addition of one or more of these intermediate alloys to the melted magnesium alloy necessarily results in a mixture of magnesium or magnesium alloy and the additive material. Thus, Xiao discloses all of the limitations of this claim element.

d. **“said additive material having a greater melting point temperature than a solidus temperature of said magnesium, [. . . said additive material including one or more metals selected from the group consisting of copper, nickel cobalt, titanium, and iron]”**

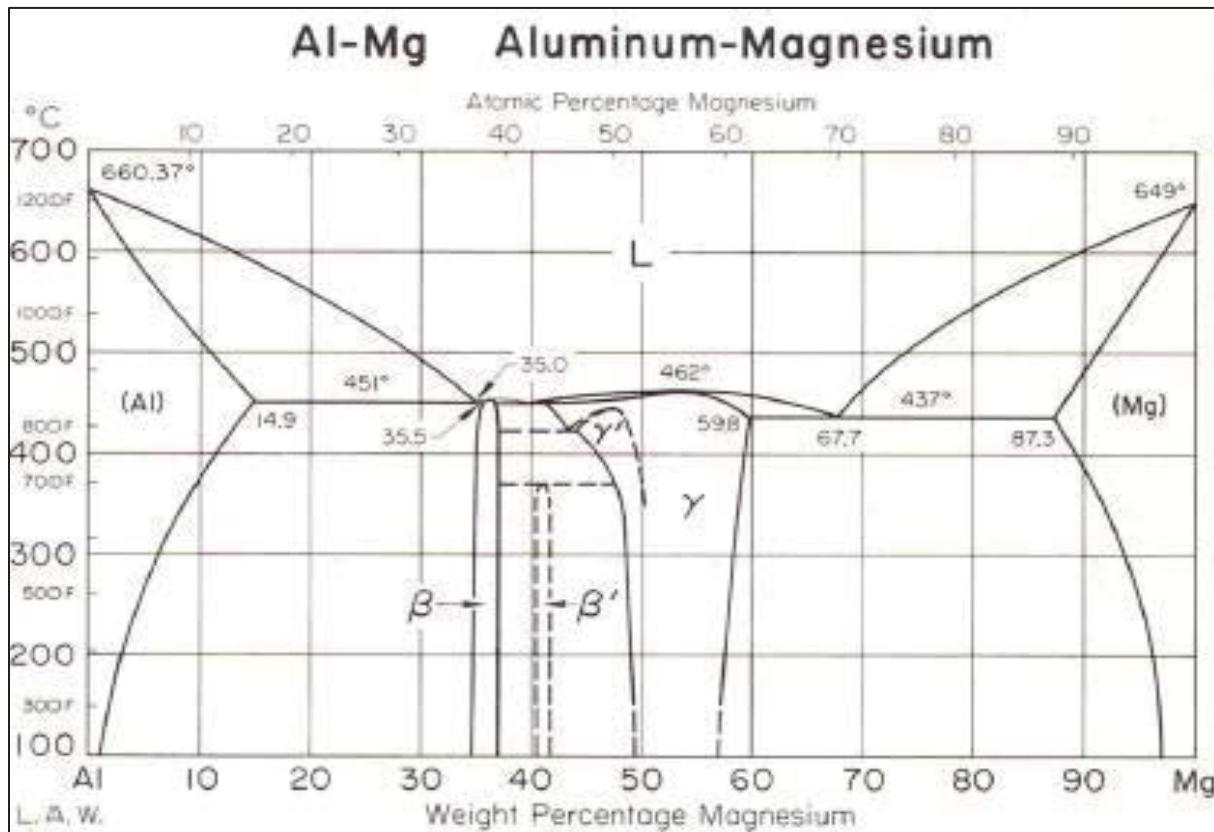
360. **All of the prior elements of Independent Claim 12 had referred to “magnesium composite” rather than just “magnesium,”** which the claim defined as comprising “a mixture

Attorney Docket No. 2189.006

Patent 10,329,653

of magnesium or magnesium alloy (and an additive material).” It is my opinion that a POSITA would have understood the reference to “magnesium” in this claim element as reference to the previous limitation of “magnesium or magnesium alloy.”

361. Using a typical phase diagram, a POSITA in April 2014 would have readily determined that the solidus temperature of magnesium is the temperature at which a metal first begins to melt during heating and not necessarily when the metal is completely liquidus.



The POSITA would also use this phase diagram to find the solidus temperature of the magnesium-aluminum alloy disclosed in Xiao to be in a range of 650°C for pure magnesium and down to 437°C for aluminum additions up to 40wt%. The solidus temperature is 437°C for the magnesium-aluminum alloys disclosed in Xiao having in the range of 13 wt% to 25 wt% aluminum.

Attorney Docket No. 2189.006

Patent 10,329,653

362. The **additive materials** disclosed in Xiao that were noted above are one or more of the **intermediate alloys: Al-Fe, Al-Ni, Al-Cu, Al-Ag, Al-Zr, Al-Ti.** *Id.* at ¶ 0023.

363. Patent Owner has taken the position that “melting point temperature” would have meant “temperature at which liquid is formed” to a POSITA in April 2014. The POSITA in April 2014 using typical phase diagrams for each of the additive materials (here the “intermediate alloys” disclosed in Xiao) would be able to readily determine the melting point temperature for each intermediate alloy. For example, the **Al-Fe** phase diagram shows a variable melting point temperature ranging from 1538°C (pure iron) down to 655°C for a eutectic temperature between about 63 wt% aluminum to near 99 wt% aluminum. The **Al-Ni** phase diagram also shows a variable melting point temperature that ranges from 1640°C down to 640°C depending upon the alloy content. The **Al-Cu** phase diagram shows a variable melting point temperature ranging from 548°C up to 1084°C depending upon the alloy mixture. A POSITA in April 2014 would have readily determined the specific melting temperature of a specific alloy mixture based on the content of these typical phase diagrams.

364. Xiao discloses a variety of **Al-Fe, Al-Ni, Al-Cu, Al-Ag, Al-Zr, Al-Ti alloy mixtures** with a variety of constituencies. For instance, Xiao specifically teaches in Example 7 that:

The composition of the alloy and the respective percentages by weight are as follows: 25% Al-10% Zn-1% Fe-0.5% Ni-0.1% Cu-0.5% Ti-0.05% Zr, and the remainder is Mg.

Xiao at ¶ 0059. Xiao teaches that the magnesium alloy of Example 7, as a whole, ends up with the maximum amount of aluminum (i.e. 25 wt%) taught as being within the invention of Xiao (see, Xiao ¶ 0026). Xiao specifically discloses--with respect to Example 7--that portions of that aluminum content are added into a magnesium-aluminum melt as an **Al-Fe intermediate alloy**,

Attorney Docket No. 2189.006

Patent 10,329,653

an **Al-Ni intermediate alloy**, an **Al-Cu intermediate alloy**, an Al-Ag intermediate alloy, an Al-Zr intermediate alloy, and an **Al-Ti intermediate alloy**. Xiao at ¶ 0060. Thus, it is necessary to prepare mass balance calculations to determine the relative concentrations of the pure magnesium and pure aluminum initially loaded into the smelting furnace in order to determine its solidus temperature.

365. First, a POSITA in April 2014, would have converted the weight percentages to grams to perform mass balances. As a result, a POSITA would have understood in April 2014 that Example 7 of Xiao includes 62.85 grams of magnesium (i.e., 100 grams - 37.15 grams (i.e., 25 grams (Al) -10 grams (Zn)- 1 gram (Fe) - 0.5 grams (Ni) - 0.1 gram (Cu) - 0.5 grams (Ti) - 0.05 grams (Zr))).

366. First, a POSITA in April 2014, would have converted the weight percentages to grams to perform mass balances. As a result, a POSITA would have understood in April 2014 that Example 7 of Xiao includes 62.85 grams of magnesium (i.e., 100 grams - 37.15 grams (i.e., 25 grams (Al) -10 grams (Zn)- 1 gram (Fe) - 0.5 grams (Ni) - 0.1 gram (Cu) - 0.5 grams (Ti) - 0.05 grams (Zr))).

367. The following intermediate alloy compositions were chosen for the initial mass balance calculations: Al-Fe (30 wt% Fe), Al-Ni (45 wt% Ni), Al-Cu (40 wt% Cu), Al-Ti (10 wt% Ti), Al-Zr (20 wt% Zr) alloy mixtures.

368. As would a POSITA in April 2014, (using the weight percentages-to-grams conversion noted above) I performed the initial mass balances.

369. Thus, using the initially selected Al-30Fe, Al-45Ni, Al-40Cu, Al-10Ti, and Al-20Zr alloys would have resulted in 7.79 grams of Aluminum being added to the overall alloy as components of the intermediate alloys (as opposed to the aluminum introduced into the ultimate

Attorney Docket No. 2189.006

Patent 10,329,653

alloy of Example 7 in the initial melt). Since Example 7 of Xiao calls for a total of 25 grams (i.e. 25 wt%) of Aluminum, 17.21 grams of Aluminum would have had to have been initially loaded into the smelting furnace in Example 7 along with the 62.85 grams of magnesium (see calculation above). Thus, the initial melt of Example 7 (using the intermediate alloys selected) would have had a composition of 78.5 wt% Mg and 21.5 wt% Al.

370. Next, a POSITA would have consulted the Mg-Al phase diagram to determine that the solidus temperature of the initial magnesium alloy (i.e. 78.5 wt% Mg and 21.5 wt% Al) of Example 7 of Xiao to be 437 °C.

371. A POSITA would also consult the relevant phase diagram to determine the melting point temperature of the various additive materials disclosed in Xiao. An aluminum-iron alloy with 30 wt% iron would have a melting point temperature of 655 °C, an aluminum-nickel alloy with 45 wt% Ni would have a melting point temperature of 854 °C, an aluminum-copper alloy with 40 wt% Cu would have a melting point temperature of 548°C, an aluminum-titanium alloy with 10 wt% Ti would have a melting point temperature of 665°C, and an aluminum-zirconium alloy with 20 wt% Zr would have a melting point temperature of 661°C. Thus, each of these additive materials from Example 7 of Xiao (with the constituencies selected above) would have had a greater melting point temperature than the solidus temperature of magnesium or the magnesium-aluminum alloy (i.e. 437°C) corresponding to 21.5 wt% aluminum which is the calculated amount of pure aluminum in pure magnesium initially loaded into the smelting furnace in Example 7. A POSITA in April 2014 would have also recognized that other compositions would have fallen within the teachings of Example 7 of Xiao as well as the other teachings of Xiao and resulted in **Al-Fe, Al-Ni, and Al-Cu intermediate alloys** having greater melting point

Attorney Docket No. 2189.006

Patent 10,329,653

temperatures than the solidus temperature of initial magnesium alloy loaded in the various Examples of Xiao. Thus, Xiao discloses all of the limitations of this claim element.

e.       **“said composite including greater than 50 wt % magnesium,”**

372. Claim 1 of Xiao discloses a “magnesium alloy, comprising the components at the weight percentages as follows: Al: 13 to 25%, Zn: 2 to 15%; **the remainder is Mg**, and a sum of the weight percentages of the components is 100%.” Xiao at Claim 1.

373. A POSITA would have understood that the total range of the non-magnesium elements disclosed in Claim 1 of Xiao is 15% (*i.e.*, 13 + 2) to 40% (*i.e.*, 25 + 15). Thus, claim 1 of Xiao would have further disclosed to a POSITA a range of magnesium of 60 wt% to 85 wt%, which range is “greater than 50 wt% magnesium” required by this limitation of the claim. Further, a POSITA would have understood that the composite of Example 7 (using the example intermediate alloys selected above) would have contained 62.85 wt% magnesium (as already calculated above), which is also greater than 50 wt% magnesium” required by this limitation of the Claim. A POSITA in April 2014 would have also recognized that the selection of other intermediate alloy compositions would have fallen within the teachings of Example 7 of Xiao as well as the other teachings of Xiao and resulted in would have resulted in magnesium composites that included greater than 50 wt% (up to about 85 wt%) magnesium. Thus, this element of the claim was disclosed by Xiao.

f.       **“said additive material constituting about 0.05 wt %-45 wt % of said magnesium composite, [. . . said additive material including one or more metals selected from the group consisting of copper, nickel cobalt, titanium, and iron]”**

374. Claim 1 of Xiao discloses a “magnesium alloy, comprising the components at the weight percentages as follows: **Al: 13 to 25%, Zn: 2 to 15%**, the remainder is Mg, and a sum of the weight percentages of the components is 100%.” Xiao at Claim 1. And Claim 2 discloses: a

Attorney Docket No. 2189.006

Patent 10,329,653

“magnesium alloy according to claim 1, further comprising the trade elements at the weight percentages as follows: **Fe: 0.1 to 5%, Cu: 0.05 to 5%, Ni: 0.05 to 5%**, Zr: 0.05 to 0.5%, **Ti: 0.05 to 0.5%**; and a sum of the weight percentages of the components is 100%.” *Id.* at Claim 2.

375. These metallic elements are found in the additive materials generally disclosed by Xiao (*i.e.*, **intermediate alloys: Al-Fe, Al-Ni, Al-Cu, Al-Ag, Al-Zr, Al-Ti**) (Xiao at ¶ 0023) and particularly described in association with Example 7 of Xiao (*Id.* at ¶¶ 0057 - 0060) for which I have already calculated (and set forth above) the specific weight percentage (ranges) of each additive material (which has been defined by the Patent Owner as “a material that is added” (see Paragraph 78 of my Declaration)) set forth in Example 7 of Xiao (*i.e.* **Al-Fe, Al-Ni, Al-Ag, Al-Cu, Al-Zr, Al-Ti** and Zinc).

376. In particular, the additive materials **aluminum-iron** with 30 wt% iron, **aluminum-nickel** with 45 wt% nickel, and **aluminum-copper** with 40 wt% copper, which each have greater melting point temperatures than the solidus temperature of magnesium or the magnesium aluminum alloy disclosed in Xiao, also fall well within the range of about 0.05 wt% to 45 wt% of the mixture. For example, the **aluminum-iron** additive constitutes 3.3 wt% of the mixture (*i.e.*, 1%/0.30); the **aluminum-nickel** additive constitutes 1.1 wt% of the mixture (*i.e.*, 0.5%/0.45); and the **aluminum-copper** additive constitutes 0.25 wt% of the mixture (*i.e.*, 0.1%/0.40). A POSITA in April 2014 would have also recognized that other intermediate alloy compositions would have fallen within the teachings of Example 7 of Xiao as well as the other teachings of Xiao and resulted in **Al-Fe, Al-Ni, and Al-Cu intermediate alloys** constituting about 0.05 wt% to 45 wt% of the magnesium composite. Accordingly, this element of the claim is disclosed by Xiao.

Attorney Docket No. 2189.006

Patent 10,329,653

- g. “said additive material having a melting point temperature that is 100°C. greater than a melting temperature of said magnesium or magnesium alloy,”

377. A POSITA would have understood “melting temperature” in this claim element to be a reference to the smelting temperature or temperature of the melt when the additive material is being added to the magnesium or magnesium alloy, not the “melting point temperature” as that measurement is used for the additive material.

378. Xiao generally discloses “loading pure magnesium and pure aluminum into a smelting furnace and **increasing the temperature to 700 to 730 °C, next loading** pure zinc and **an [sic] intermediate alloys of trace element components into** a resulting magnesium-aluminum alloy melt after melting and increasing the temperature to 740 to 780°C . . . .” Xiao at ¶¶ 0021–0022; see also *Id.* at Claim 5 (“the **melting temperature of the pure magnesium and pure aluminum is from 700 to 730 °C.**”)

379. Examples 1-7 of Xiao teach different **melting or smelting temperatures for the magnesium-aluminum alloy, ranging from a low of 700°C** in Example 1 to a high of **730 °C** in Examples 2, 5, and 7. Xiao ¶¶ 0034–0060. In each Example, “[a]fter melting” the intermediate alloy are “added into the resulting magnesium-aluminum alloy melt, and at the same time the temperature is raised” to a different temperature depending on the example. *Id.*

380. The **additive materials** noted above are one or more of the **intermediate alloys: Al-Fe, Al-Ni, Al-Cu, Al-Ag, Al-Zr, Al-Ti.** *Id.* at ¶ 0023. As I noted in Paragraph 78 of Declaration, Patent Owner has taken the position that “melting point temperature” would have meant “temperature at which liquid is formed” to a POSITA in April 2014.

381. As already noted in Paragraph 152 of my Declaration (above), based on the typical phase diagrams, a POSITA in April 2014 would have recognized that **Al-Fe** had a variable melting

Attorney Docket No. 2189.006

Patent 10,329,653

point temperature ranging from 1538°C (pure iron) down to 652°C for a eutectic point near 99 wt% aluminum; **Al-Ni** had a variable melting point temperature that ranges from 1640°C down to 640°C (depending upon the alloy mixture); and **Al-Cu** had a variable melting point temperature ranging from 548°C up to 1084°C (depending upon the alloy mixture). A POSITA in April 2014 would have been able to determine the specific melting point temperature of a specific alloy mixture based on the content of these typical phase diagrams.

382. Example 7 specifically discloses a melting or smelting temperature of 730°C. Xiao at ¶ 0060. I specifically analyzed Example 7 of Xiao above to determine (using particular intermediate alloy compositions) the melting point temperatures of the intermediate alloys added. As a result, it is my opinion that a POSITA in April 2014 would recognize from Example 7 of Xiao that at least an aluminum-nickel additive having 45 wt% nickel has a melting point temperature of 854°C which is 100°C greater than the melting or smelting temperatures of the magnesium aluminum alloy used in Example 7 (*i.e.*, 730°C) and otherwise taught generally by Xiao (*i.e.*, 700-730°C). A POSITA in April 2014 would have also recognized that other intermediate alloy compositions could have fallen within the teachings of Example 7 of Xiao and resulted in **Al-Fe**, **Al-Ni**, and **Al-Cu intermediate alloys** that would have had melting point temperatures that were 100°C greater than the smelting temperatures of the magnesium alloy generally taught by Xiao (*i.e.*, 700-730°C).<sup>15</sup> Thus, Xiao discloses all of the elements of this claim limitation.

---

<sup>15</sup> It is worth pointing out—although I would disagree with either notion—that if Patent Owner argues either that “melting temperature” was a typographical error (which I doubt given the other claims that say “melting point temperature” and the numerous Certificates of Correction that did not address this issue) or simply argues that “melting temperature of said magnesium or magnesium alloy” means “melting point temperature of said magnesium or magnesium alloy,” not only would the Al-Ni intermediate alloy with 45 wt% nickel exceed the *melting point temperature* of pure magnesium (*i.e.*, 650°C) and the *melting point temperature of the initially loaded*

Attorney Docket No. 2189.006

Patent 10,329,653

- h. “said **additive material** includes one or more metals selected from the group consisting of copper, nickel, cobalt, titanium, and iron,”

383. The **intermediate alloys Al-Cu, Al-Ti, Al-Fe, and Al-Ni** exhibit specific chemistry ranges with melting point temperatures that are both (i) greater than the solidus temperature of the magnesium aluminum alloy disclosed in Xiao and (ii) 100°C greater than the melting/smelting temperature of that alloy (disclosed in Xiao to be 700-730°C). For example, in Example 7 of Xiao, an **aluminum-nickel** additive having 45 wt% nickel used in the material balance calculations, has a melting point temperature of 854°C determined from the Al-Ni phase diagram, the melting point temperature being 100°C greater than the melting temperature of that Example 7 of Xiao (i.e., 730°C). A POSITA in April 2014 would have recognized that an **Al-Ni intermediate alloy** of Example 7 of Xiao contained **nickel, which is a metal**. A POSITA in April 2014 would have also recognized that the **Al-Cu, Al-Ti, and Al-Fe intermediate alloys** would have also met the other temperature and weight percentage limitations of this claim, and would have further recognized they respectively included **copper, titanium and iron each of which are metals**. Thus, Xiao discloses the use of one or more additive materials of one or more metals including copper, nickel, titanium, and/or iron.

- i. “at least a portion of said **additive material** remaining **unalloyed additive material**,”

384. Xiao discloses that “[e]lements such as Fe, Cu, Ni, Ag, etc., in the magnesium alloy can form a large number of intermetallic composite micro-particles.” *Id.* at ¶ 0026.

---

**magnesium-aluminum alloy** (i.e., 438°C) of Example 7 using the constituents discussed in Paragraphs 114-118 of my Declaration (i.e., a composition of 78.5 wt% Mg and 21.5 wt% Al), by at least 100°C, but also the Al-Cu, Al-Fe, Al-Ti and Al-Zr intermediate alloys, having the constituents set forth in those Paragraphs of my Declaration would similarly exceed that limitation.

Attorney Docket No. 2189.006

Patent 10,329,653

385. From the foregoing elements of the claim we know that the potential claimed “additive material” disclosed by Xiao is limited to Fe, Cu, Ni, and Ag. However, Xiao does not disclose whether any of the additive material remains “unalloyed” (which has been defined by the Patent Owner as “portion of the additive material that does not form a new compound” (see Paragraph 78 of my Declaration)).

386. Hassan, on the other hand, specifically discloses that magnesium composites containing additive materials, such as nickel, may remain unreacted (i.e. does not form a new compound). In particular, Hassan states that:

[s]evere reaction between magnesium melt and nickel particulates during DMD processing led to the reduction of the particulate size and formation of Mg<sub>2</sub>Ni intermetallics [13]. **The results of quantitative determination of unreacted nickel** (see Table I), microstructural characterization illustrating the presence of reaction products (see Fig. 1) and XRD results (see Table II) showing the presence of Mg<sub>2</sub>Ni supports the experimental observations.

**TABLE I** Results of density and porosity measurements

Material	Reinforcement in composites				Density (g/cm <sup>3</sup> )
	(wt%)	(vol%) <sup>a</sup>	(vol%) <sup>b</sup>	Size <sup>c</sup>	
Mg	—	—	—	—	1.7395 ± 0.0005
Mg/7.3Ni <sub>P</sub>	7.3	1.5	0.1	2.7 ± 0.6	1.9046 ± 0.0038
Mg/14.0Ni <sub>P</sub>	14.0	3.1	1.3	2.5 ± 0.4	2.0677 ± 0.0002
Mg/24.9Ni <sub>P</sub>	24.9	6.1	4.3	9.0 ± 4.6.	2.3834 ± 0.0092

<sup>a</sup>Computed by using the total amount of Ni retained in composite samples.

<sup>b</sup>Indicates the amount of Ni in the unreacted form present in composite samples.

<sup>c</sup>Result of cumulative image analysis conducted on fifteen representative SEM micrographs for each composite.

**TABLE II Results of X-ray diffraction studies and coefficient of thermal expansion analysis**

Materials	Number of matching peaks					CTE ( $\times 10^{-6}/^{\circ}\text{C}$ )
	Mg	Ni	$\text{Mg}_2\text{Ni}$	$\text{NiO}$	$\text{Ni}_2\text{O}_3$	
Mg	9 [3]	—	—	—	—	$28.60 \pm 0.07$
Mg/7.3Ni <sub>p</sub>	6 [3]	—	6 [2]	—	—	$27.54 \pm 0.26$
Mg/14.0Ni <sub>p</sub>	5 [3]	—	8 [2]	—	—	$26.35 \pm 0.09$
Mg/24.9Ni <sub>p</sub>	6 [3]	2 [1]	16 [2]	1 [1]	1	$20.75 \pm 0.56$

[ ] indicates the number of main peaks matched.

Hassan at 2472 (emphasis added).

387. Hassan (like Xiao) discloses the fabrication of magnesium based materials or alloys and teaches that there are benefits to reinforcing these materials or alloys with other metals or metal alloys to improve the physical properties of the resulting magnesium composite. Hassan also recognizes that type of processing, type, size, and volume fraction of reinforcement are “common factors governing the end properties of a metal matrix composite[.]” Hassan at 2467. Xiao similarly teaches in the context of its smelt casted magnesium alloy, the use of reinforcing metals or metal alloys to enhance corrosion performance and/or improve the compressive strength of the formed magnesium material. Xiao at ¶ 0026. In particular, Xiao noted that existing magnesium alloys (such as AZ91D) had low compressive strength and slow decomposition rates, making them unsuitable as material for, for example, a tripping ball used in oil well drilling. *Id* at 0003.

388. Unlike Xiao, which does not explicitly mention the possibility of there being unreacted additives, Hassan specifically provides experimental data showing—under similar

Attorney Docket No. 2189.006

Patent 10,329,653

conditions of magnesium composite fabrication as those in Xiao—that some of the additives, particularly nickel, will remain unreacted and unalloyed.

389. Given this motivation to combine Xiao with Hassan, it is my opinion that a POSITA in April 2014 would have recognized that these two references disclose this limitation of the claim.

- j. **“said magnesium composite including *in situ* precipitation of galvanically-active intermetallic phases that includes said unalloyed additive material,”**

390. Xiao discloses that “[e]lements such as Fe, Cu, Ni, Ag, etc., in the magnesium alloy **can form a large number of intermetallic composite micro-particles**, which can improve the corrosion performance of the magnesium alloy, thereby promoting the decomposition of the magnesium alloy.” *Id.* at ¶ 0026, ll. 15–18. A POSITA in August 2014 would have understood this disclosure of “intermetallic composite micro-particles” to be a reference to intermetallic precipitate.

391. As noted above, Hassan discloses that added nickel (Ni) will remain unalloyed (i.e. unreacted) under certain conditions.

392. A POSITA reading the Xiao and Hassan references in April 2014 would have recognized that the precipitation of galvanically-active intermetallic phases (“micro-batteries”) are necessarily generated by the *in situ* reaction between magnesium and the disclosed metal additives forming *in situ* precipitate on or with any unalloyed or unreacted additive material. Thus, Xiao together with Hassan disclosed this element of the claim.

- k. **“said magnesium composite has a dissolution rate of at least 5 mg/cm<sup>2</sup>/hr. in 3 wt % KCl water mixture at 90°C.”**

393. A POSITA in April 2014 would have understood the English translation of Xiao to disclose a table of dissolution rates (“decomposition rates”) because the units of measurement in

Attorney Docket No. 2189.006

Patent 10,329,653

that table are units regularly used by POSITAs to express measurements of dissolution. In particular, Xiao provides dissolution rates for its various examples, as follows:

	Room temperature tensile strength $\sigma_b$ (MPa)	Decomposition rate at 70 °C in 3% KCl solution (g.cm <sup>-2</sup> .h <sup>-1</sup> )	Decomposition rate at 93 °C in 3% KCl solution (g.cm <sup>-2</sup> .h <sup>-1</sup> )
Comparative example 1	232	0.00026	0.0005
Example 1	360	0.035	0.074
Example 2	385	0.015	0.045
Example 3	410	0.013	0.036
Example 4	375	0.034	0.058
Example 5	392	0.025	0.048
Example 6	365	0.021	0.063
Example 7	387	0.036	0.057

Xiao at ¶ 0064.

394. Xiao provides dissolution rates in g/cm<sup>2</sup>/hr, while the claims of the '653 Patent express the claimed dissolution rate in mg/cm<sup>2</sup>/hr. A POSITA in April 2014 would have known that converting from g/cm<sup>2</sup>/hr to mg/cm<sup>2</sup>/hr, merely requires multiplication of the numbers disclosed in this table from Xiao by 1,000.

395. Thus, the dissolution rates disclosed in Xiao convert to:

	Decomposition rate at 70°C in 3% KCl solution mg/cm <sup>2</sup> /hr	Decomposition rate at 93°C in 3% KCl solution mg/cm <sup>2</sup> /hr
Comparative example 1	0.26	0.5

Attorney Docket No. 2189.006

Patent 10,329,653

Example 1	35	74
Example 2	15	45
Example 3	13	36
Example 4	34	58
Example 5	25	48
Example 6	21	63
Example 7	36	57

396. Each of the seven examples (excluding the comparative [prior art] example) provided in Xiao therefore discloses a dissolution rate of “at least 5 mg/cm<sup>2</sup>/hr in 3 wt.% KCl water” at both 70°C or 93°C. Given that the disclosed dissolution rates of Xiao both above and below 90°C are well above the claimed floor of “at least 5 mg/cm<sup>2</sup>/hr” (i.e. at least three times overall and for Example 7 at least seven times) it is my opinion that a POSITA in April 2014 would have recognized the dissolution rate of the examples in Xiao at 93°C to also be necessarily well above the claimed floor of “at least of 5 mg/cm<sup>2</sup>/hr.” Consequently, this element of Claim 1 is also disclosed by Xiao.

397. Accordingly, as set forth in the immediately foregoing paragraphs, it is my opinion that Xiao, in view of Hassan’s disclosure that Nickel would remain unalloyed, would have fully disclosed each and every element of Claim 12 of the ‘653 Patent to a POSITA in or before April 2014 in accordance with Patent Owner’s definitions (set forth in Paragraph 78 of my Declaration).

**3. Dependent Claim 13 -- “The magnesium composite as defined in claim 12, wherein said additive material is added to said magnesium or magnesium alloy while said magnesium or magnesium alloy is at a temperature that is above said solidus temperature of said magnesium and a temperature that is less**

Attorney Docket No. 2189.006

Patent 10,329,653

**than a melting point of said additive material to form said mixture.”**

398. As set forth above, it is my opinion that Xiao in combination with Hassan would have disclosed all elements of Claim 12 to a POSITA in April 2014.

399. Xiao discloses “first loading pure magnesium and pure aluminum into a smelting furnace, **next loading** pure zinc and **an [sic] intermediate alloys** of trace element components into a resulting magnesium-aluminum alloy melt **after melting.**” Xiao at Claim 4. Xiao further discloses “loading pure magnesium and pure aluminum into a smelting furnace and increasing the temperature to 700 to 730 °C, **next loading** pure zinc and **an [sic] intermediate alloys** of trace element components into a resulting magnesium-aluminum alloy melt **after melting.**” *Id.* at ¶ 0022. Using a typical phase diagram, a POSITA in April 2014 would have readily determined that the solidus temperature of magnesium is 650°C. The POSITA would also use the phase diagram to find the solidus temperature of the magnesium-aluminum alloy disclosed by Xiao to be between 437-650°C.

400. Xiao further discloses that the pure Zinc and intermediate alloys are loaded into the magnesium-aluminum melt, which is at a temperature of 700 to 730 °C, and then “increasing the temperature to 740 to 780°C.” Xiao at ¶ 0022.

401. Next, given the limitation of Independent Claim 12 (addressed above) that “said additive material having a melting point temperature that is 100°C greater than a melting temperature of said magnesium or magnesium alloy,” any additive material that meets the limitations of Claim 12 will also necessarily meet the limitation that the “additive material is added to said . . . magnesium alloy while said . . . magnesium alloy is at . . . a temperature that is less than a melting point of said additive material” given Xiao’s disclosure that the intermediate alloys are added to the magnesium-aluminum melt at a temperature of 700 to 730 °C, (and then

Attorney Docket No. 2189.006

Patent 10,329,653

increased to 740 to 780°C). As noted above in Paragraph 381 of my Declaration (above), the **intermediate alloys Al-Cu, Al-Ti, Al-Fe, and Al-Ni** exhibit specific chemistry ranges with melting point temperatures that are both (i) greater than the solidus temperature of the magnesium aluminum alloy disclosed in Xiao and (ii) 100°C greater than the melting/smelting temperature of that alloy (disclosed in Xiao to be 700-730°C) and, thus also meets the added limitations of this dependent claim. Thus, Xiao discloses all of the limitations of Dependent Claim 13.

#### 4. Dependent Claim 71

- a. **“The magnesium composite as defined in claim 49, wherein at least a portion of said additive material remains unalloyed additive material in said magnesium composite.”**

402. As set forth above, it is my opinion that Xiao would have disclosed all elements of Claim 49 to a POSITA in April 2014.

403. Xiao discloses that “[e]lements such as **Fe, Cu, Ni, Ag, etc.**, in the magnesium alloy can form a large number of intermetallic composite micro-particles.” *Id.* at ¶ 0026.

404. From the foregoing elements of the claim we know that the potential claimed “additive material” disclosed by Xiao is limited to Cu, Ni, Ti, Fe, and Co. However, Xiao does not disclose whether any of the additive material remains “unalloyed” (which has been defined by the Patent Owner as “portion of the additive material that does not form a new compound” (see Paragraph 78 of my Declaration)).

405. Hassan, on the other hand, specifically discloses that magnesium composites containing additive materials, such as nickel, may remain unreacted (i.e. does not form a new compound). In particular, Hassan states that:

[s]evere reaction between magnesium melt and nickel particulates during DMD processing led to the reduction of the particulate size and formation of Mg<sub>2</sub>Ni intermetallics [13]. **The results of quantitative determination of unreacted**

Attorney Docket No. 2189.006

Patent 10,329,653

**nickel** (see Table I), microstructural characterization illustrating the presence of reaction products (see Fig. 1) and XRD results (see Table II) showing the presence of Mg<sub>2</sub>Ni supports the experimental observations.

**TABLE I** Results of density and porosity measurements

Material	Reinforcement in composites				Density (g/cm <sup>3</sup> )
	(wt%)	(vol%) <sup>a</sup>	(vol%) <sup>b</sup>	Size <sup>c</sup>	
Mg	—	—	—	—	1.7395 ± 0.0005
Mg/7.3Ni <sub>P</sub>	7.3	1.5	0.1	2.7 ± 0.6	1.9046 ± 0.0038
Mg/14.0Ni <sub>P</sub>	14.0	3.1	1.3	2.5 ± 0.4	2.0677 ± 0.0002
Mg/24.9Ni <sub>P</sub>	24.9	6.1	4.3	9.0 ± 4.6.	2.3834 ± 0.0092

<sup>a</sup>Computed by using the total amount of Ni retained in composite samples.

<sup>b</sup>Indicates the amount of Ni in the unreacted form present in composite samples.

<sup>c</sup>Result of cumulative image analysis conducted on fifteen representative SEM micrographs for each composite.

**TABLE II** Results of X-ray diffraction studies and coefficient of thermal expansion analysis

Materials	Number of matching peaks					CTE (× 10 <sup>-6</sup> /°C)
	Mg	Ni	Mg <sub>2</sub> Ni	NiO	Ni <sub>2</sub> O <sub>3</sub>	
Mg	9 [3]	—	—	—	—	28.60 ± 0.07
Mg/7.3Ni <sub>P</sub>	6 [3]	—	6 [2]	—	—	27.54 ± 0.26
Mg/14.0Ni <sub>P</sub>	5 [3]	—	8 [2]	—	—	26.35 ± 0.09
Mg/24.9Ni <sub>P</sub>	6 [3]	2 [1]	16 [2]	1 [1]	1	20.75 ± 0.56

[ ] indicates the number of main peaks matched.

Hassan at 2472 (emphasis added).

406. Hassan (like Xiao) discloses the fabrication of magnesium based materials or alloys and teaches that there are benefits to reinforcing these materials or alloys with other metals or metal alloys to improve the physical properties of the resulting magnesium composite. Hassan also recognizes that type of processing, type, size, and volume fraction of reinforcement are “common

Attorney Docket No. 2189.006

Patent 10,329,653

factors governing the end properties of a metal matrix composite[].” Hassan at 2467. Xiao similarly teaches in the context of its smelt casted magnesium alloy, the use of reinforcing metals or metal alloys to enhance corrosion performance and/or improve the compressive strength of the formed magnesium material. Xiao at ¶ 0026. In particular, Xiao noted that existing magnesium alloys (such as AZ91D) had low compressive strength and slow decomposition rates, making them unsuitable as material for, for example, a tripping ball used in oil well drilling. *Id* at 0003.

407. Unlike Xiao, which does not explicitly mention the possibility of there being unreacted additives, Hassan specifically provides experimental data showing--under similar conditions of magnesium composite fabrication as those in Xiao--that some of the additives, particularly nickel, will remain unreacted and unalloyed.

408. Given this motivation to combine Xiao with Hassan, it is my opinion that a POSITA in April 2014 would have recognized that these two references disclose this limitation of the claim. Consequently, all of the limitations of Dependent Claim 71 are taught by Xiao in combination with Hassan.

## VIII. CONCLUSION

I hereby declare under penalty of perjury under the law of the United States of America that the foregoing is true and correct, and that all statements made of my own knowledge are true and that all statements made on information and belief are believed to be true. I understand that willful false statements are punishable by fine or imprisonment or both. 18 U.S.C. § 1001.



Dated: July 6, 2021

By:

Dana J. Medlin, Ph.D., P.E., FASM

60002

Date:

9/11/2020

## Xiao Example #7 - Mass Balance

Assume 100 gr

1] 25% Al

10% Zn

1% Fe

0.5% Ni

0.1% Cu

0.5% Ti

0.05% Zr

37.15% Alloys

62.85% Mg gr (Balance)

100.00 Total

① Mg (m.p. 650°C) + Al (m.p. 660°C) heat to 730°C

② Add: Al-Fe + Al-Ni + Al-Cu + ~~Al-Ag~~ + Al-Zr +  
Al-Ti + pure ZnRaised to 760°C, Then lowered to 730°C,  
degassed then cooled to 710°C, Then cooled.

Al-Fe 1% Fe

(70% Al + 30% Fe)

m.p = 655°C

$$\frac{x \text{ gr Al}}{1.0 \text{ gr Fe}} = \frac{0.70 \text{ gr Al}}{0.30 \text{ gr Fe}}$$

x = 2.33 gr Al

60002

Prepared by: DT Merlin  
 Date: 9/11/2020

2

Al-Ni

0.5% Ni

(55% Al + 45% Ni)

m.p. = 854 °C

$$\frac{x \text{ gr Al}}{0.5 \text{ gr Ni}} = \frac{0.55 \text{ gr Al}}{0.45 \text{ gr Ni}}$$

$$x = 0.61 \text{ gr Al}$$

0.1% Cu

(60% Al + 40% Cu)

m.p. = 548 °C

$$\frac{x \text{ gr Al}}{0.1 \text{ gr Cu}} = \frac{0.60 \text{ gr Al}}{0.40 \text{ gr Cu}}$$

$$x = 0.15 \text{ gr Al}$$

0.5% Ti

(90% Al + 10% Ti)

m.p. = 665 °C

$$\frac{x \text{ gr Al}}{0.5 \text{ gr Ti}} = \frac{0.90 \text{ gr Al}}{0.10 \text{ gr Ti}}$$

$$x = 4.5 \text{ gr Al}$$

0.05% Zr

(80% Al + 20% Zr)

m.p. 661 °C

$$\frac{x \text{ gr Al}}{0.05 \text{ gr Zr}} = \frac{0.80 \text{ gr Al}}{0.20 \text{ gr Zr}}$$

$$x = 0.20 \text{ gr Al}$$

Prepared by: Dr. Mehta

Date: 9/11/2020

3

Zn added as pure 10%

Total Al added as intermediate alloy:

$$2.33_{\text{gr}} + 0.61_{\text{gr}} + 0.15_{\text{gr}} + 4.5_{\text{gr}} + 0.20_{\text{gr}} = 7.79_{\text{gr}}$$

## Mass Balance:

$$25 \text{ gr Al} - 7.79 \text{ gr} = 17.21 \text{ gr}$$

↑ from Step ②

Start: ① 17.21 gr Al + 62.85 gr Mg and melt.  
Subtotal: 80.06 gr at 730°C

② Add Intermediate alloys

$$\left. \begin{array}{l} 1.0 \text{ gr Fe} + 2.33 \text{ gr Al} \quad (\text{Al-Fe}) \\ 0.5 \text{ gr Ni} + 0.61 \text{ gr Al} \quad (\text{Al-Ni}) \\ 0.1 \text{ gr Cu} + 0.15 \text{ gr Al} \quad (\text{Al-Cu}) \\ 0.5 \text{ gr Ti} + 4.5 \text{ gr Al} \quad (\text{Al-Ti}) \\ 0.05 \text{ gr Zr} + 0.20 \text{ gr Al} \quad (\text{Al-Zr}) \\ 10 \text{ gr Zn} \end{array} \right\} 9.94 \text{ gr}$$

$$80.06\text{gr} + 9.94\text{gr} + 10\text{gr} = 100.0\text{gr}$$

The invention disclosesA light and pressure-proof fast-decomposed cast magnesium alloy~~which~~ can be used as a tripping ball material for a multi-stage sliding sleeve staged-fracturing technique. The light and pressure-proof fast-decomposed cast magnesium alloy is prepared from composed of the following components: 13-to 25% of Al, 2-to 15% of Zn, 0.1-to 5% of Fe, 0.05-to 5% of Cu, 0.05-to 5% of Ni, 0-to 5% of Ag, 0.05-to 0.5% of Zr, 0.05-to 0.5% of Ti, and the balance of remainder is Mg. The preparation method comprises the following steps-~~of~~: weighing the respective components according to the designed component ratioproportions of the magnesium alloy; firstly, putting~~then first loading~~ pure magnesium and pure aluminum into a smelting furnace, putting~~next loading~~ pure zinc and an intermediate alloyalloys of the other components into a magnesium-aluminum alloy melt after melting; increasing the temperature for melting, refining, degassing, waiting for a while and stewing after warming to melt; then casting in a protective atmosphere. The light and pressure-proof fast-decomposed cast magnesium alloy of the present invention is reasonable in component ratio, and simple in craft~~the production~~ process; the light and pressure-proof fast-decomposed cast magnesium alloy can be obtained by controlling the ingredientscomponents of the alloy; the alloy decomposition performance exceeds that of the~~a~~ traditional cast magnesium alloy; the demands of the multi-stage sliding sleeve staged-fracturing technique on the decomposition performance of the tripping ball material can be met; industrialized application can be achieved; application of the magnesium alloy in the field of exploitation of petroleum and gas is thus expanded.

<b>INFORMATION DISCLOSURE STATEMENT BY APPLICANT</b>	Complete if Known	
	Application No.	
	Filing Date	Herewith
	First Named Inventor	Brian P. Doud
	Art Unit	
	Examiner Name	
	Attorney Docket No.	2189.006

<b>U. S. PATENT APPLICATION DOCUMENTS</b>					
EXAM INIT.	Cite No. <sup>1</sup>	Patent Number Number-Kind Code <sup>2</sup> (if known)	Name of Patentee or Applicant of Cited Document	Issue Date MM/DD/YYYY	
	P01	5,476,632	Shivanath et al.	12/19/1995	
<b>U. S. PATENT APPLICATION PUBLICATION DOCUMENTS</b>					
EXAM INIT.	Cite No. <sup>1</sup>	Publication Number Number-Kind Code <sup>2</sup> (if known)	Name of Patentee or Applicant of Cited Document	Publication Date MM/DD/YYYY	
<b>FOREIGN PATENT DOCUMENTS</b>					
EXAM INIT.	Cite No. <sup>1</sup>	Foreign Document No. <sup>3</sup> Country Code <sup>4</sup> - Kind Code <sup>5</sup> (if known)	Name of Patentee or Applicant of Cited Document	Date of Publication of Cited Document MM-DD-YYYY	T <sup>6</sup>
	F01	CN103343271A (Chinese-language accompanied by an English language abstract)	Xiao et al.	10/09/2013	
	F02	CN 103343271 (Certified English Language Translation)	Xiao et al.	10/09/2013	
<b>NON-PATENT DOCUMENTS</b>					
EXAM INIT.	Cite No. <sup>1</sup>	Include name of the author (in CAPITAL LETTERS), title of the article (when appropriate), title of the item (book magazine, journal, serial, symposium, catalog, etc.), date, page(s), volume-issue number(s), publisher, city and/or country where published.			
	NPL01	HASSAN, S.F., GUPTA, M., Development of High Strength Magnesium Based Composites Using Elemental Nickel Particulates as Reinforcement., <i>Journal of Materials Science</i> 37, 2467–2474 (2002).			
	NPL02	USPTO, Image File Wrapper for US Provisional Patent Application 61982425, filed 04/18/2014.			
	NPL03	HAI ZHI YE, XING YANG, LIU, Review of Recent Studies in Magnesium Matrix Composters, <i>Journal of Materials Science</i> 39 (2004).			
	NPL04	HEMANTH, Joel, Fracture Behavior of Cryogenically solidified aluminum-alloy reinforced with Nano-ZrO <sub>2</sub> Metal Matrix Composites (CNMMCs). <i>Journal of Chemical Engineering and Materials Science</i> Vol. 2(8), August 2011.			
	NPL05	SHAW, Barbara A., Corrosion Resistance of Magnesium Alloys, Pennsylvania State University. <i>ASM Handbook, Volume 13A Corrosion: Fundamentals, Testing, and Protection</i>			
	NPL06	HENAGER, Charles H. Al-Fe Diagram, Hydrogen Permeation Barrier Coatings, Materials for the Hydrogen Economy, Chapter 8, CRC Press 2007, Editors: R. H. Jones, G. J. Thomas, pp 181-190.			
	NPL07	LASZLO J. KECSKES ET AL., Al-Ni Diagram, Army Research Laboratory ARL-TR-5507, April 2011, ACombustion Synthesis Reaction Behavior of Cold-Rolled Ni/Al and Ti/Al Multilayers. p. 5.			

Sheet 2 of 2

<b>INFORMATION DISCLOSURE STATEMENT BY APPLICANT</b>	Complete if Known	
	Application No.	
	Filing Date	Herewith
	First Named Inventor	Brian P. Doud
	Art Unit	
	Examiner Name	
	Attorney Docket No.	2189.006

	NPL08	Al-Cu Diagram, <a href="https://sites.google.com/site/eampotentials/Home/AlCu">https://sites.google.com/site/eampotentials/Home/AlCu</a> downloaded June 20, 2020
	NPL09	MCALISTER, A. J., Al-Ag Diagram, Bull. Alloy Phase Diagrams, 8(6):526-533, 1987.
	NPL10	PROFESSOR MIKE ASHBY, Cu-Ni Phase Diagram. Teach Yourself Phase Diagrams and Phase Transformations, 5th Edition, March 2009 Cambridge.
	NPL11	Illustrated portion of Cu-Ni Chart Phase Diagram. Materials Science and Engineering, 6th Edition by Callister and Rethwisch, 2003.
	NPL12	Ag-Cu Binary Phase Diagram. ASM Metals Handbook, 8th Edition, Vol. 8, Metallography, Structures and Phase Diagrams, 1973.
	NPL13	Mg-Pb Phase Diagram. ASM Metals Handbook, 8th Edition, Vol. 8, Metallography, Structures and Phase Diagrams, 1973.
	NPL14	Al-Mg Phase Diagram. ASM Metals Handbook, 8th Edition, Vol. 8, Metallography, Structures and Phase Diagrams, 1973.
	NPL15	Mg-Ni Diagram. ASM Metals Handbook, 8th Edition, Vol. 8, Metallography, Structures and Phase Diagrams, 1973.
	NPL16	JONES, D.A.; Principles and Prevention of Corrosion, 2nd Ed., 1996, p. 170 - Galvanic Series Table; 1996.

**Examiner Signature:****Date Considered:**

\*EXAMINER: Initial if reference considered, whether or not citation is in conformance with MPEP 609. Draw line through citation if not in conformance and not considered. Include copy of this form with next communication to applicant.

1 Applicant's unique citation designation number.

2 See Kinds Codes of USPTO Patent Documents at [www.uspto.gov](http://www.uspto.gov) or MPEP 901.04.

3 Enter Office that issued the document, by the two-letter code (WIPO Standard ST.3).

4 For Japanese patent documents, the indication of the year of the reign of the Emperor must precede the serial number of the patent document.

5 Kind of document by the appropriate symbols as indicated on the document under WIPO Standard ST.16 if possible.

6 Applicant is to place a check mark here if English language Translation is attached.



US010329653B2

(12) **United States Patent**  
**Doud et al.**

(10) **Patent No.:** US 10,329,653 B2  
(45) **Date of Patent:** \*Jun. 25, 2019

(54) **GALVANICALLY-ACTIVE IN SITU FORMED PARTICLES FOR CONTROLLED RATE DISSOLVING TOOLS**

(71) Applicant: **Terves Inc.**, Euclid, OH (US)

(72) Inventors: **Brian P. Doud**, Cleveland Heights, OH (US); **Nicholas J. Farkas**, Euclid, OH (US); **Andrew J. Sherman**, Mentor, OH (US)

(73) Assignee: **Terves Inc.**, Euclid, OH (US)

(\* ) Notice: Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 29 days.

This patent is subject to a terminal disclaimer.

(21) Appl. No.: **15/641,439**

(22) Filed: **Jul. 5, 2017**

(65) **Prior Publication Data**

US 2017/0298492 A1 Oct. 19, 2017

**Related U.S. Application Data**

(62) Division of application No. 14/689,295, filed on Apr. 17, 2015, now Pat. No. 9,903,010.  
(Continued)

(51) **Int. Cl.**  
**C22C 1/02** (2006.01)  
**C22F 1/06** (2006.01)  
(Continued)

(52) **U.S. Cl.**  
CPC ..... **C22F 1/06** (2013.01); **C22C 1/02** (2013.01); **C22C 23/02** (2013.01); **C22C 23/00** (2013.01)

(58) **Field of Classification Search**  
CPC ... C22F 1/06; C22C 23/02; C22C 1/02; C22C 23/00

See application file for complete search history.

(56) **References Cited**

**U.S. PATENT DOCUMENTS**

3,180,728 A	4/1965	Pryor et al.
3,445,731 A	5/1969	Saeki et al.

(Continued)

**FOREIGN PATENT DOCUMENTS**

CA	2886988	10/2015
CN	101381829	3/2009

(Continued)

**OTHER PUBLICATIONS**

Sigworth et al. "Grain Refinement of Aluminum Castings Alloys" American Foundry Society; Paper 07-67; pp. 5-7 (2007).  
(Continued)

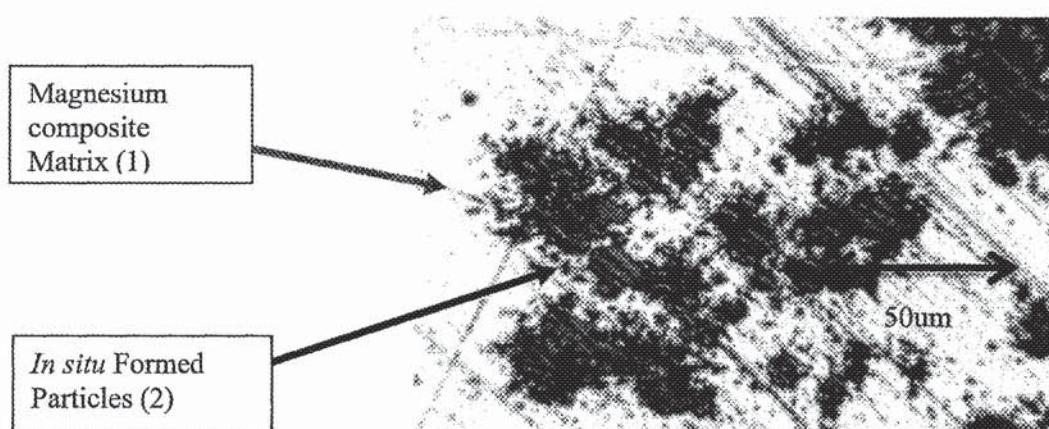
*Primary Examiner* — Anthony J Zimmer

(74) *Attorney, Agent, or Firm* — Fay Sharpe LLP

(57) **ABSTRACT**

A castable, moldable, and/or extrudable structure using a metallic primary alloy. One or more additives are added to the metallic primary alloy so that in situ galvanically-active reinforcement particles are formed in the melt or on cooling from the melt. The composite contain an optimal composition and morphology to achieve a specific galvanic corrosion rate in the entire composite. The in situ formed galvanically-active particles can be used to enhance mechanical properties of the composite, such as ductility and/or tensile strength. The final casting can also be enhanced by heat treatment, as well as deformation processing such as extrusion, forging, or rolling, to further improve the strength of the final composite over the as-cast material.

**78 Claims, 4 Drawing Sheets**



## US 10,329,653 B2

Page 2

Related U.S. Application Data						
(60)	Provisional application No. 61/981,425, filed on Apr. 18, 2014.		2006/0207387 A1	9/2006	Soran et al.	
			2006/0278405 A1	12/2006	Turley	
(51)	<b>Int. Cl.</b>		2007/0181224 A1	8/2007	Marya et al.	
	<i>C22C 23/00</i>	(2006.01)	2008/0041500 A1	2/2008	Bronfin	
	<i>C22C 23/02</i>	(2006.01)	2008/0149345 A1	6/2008	Marya et al.	
(56)	<b>References Cited</b>		2008/0175744 A1	7/2008	Motegi	
	U.S. PATENT DOCUMENTS		2009/0116992 A1	5/2009	Lee	
			2009/0226340 A1	9/2009	Marya	
			2010/0119405 A1	5/2010	Okamoto et al.	
			2010/0161031 A1	6/2010	Papirova et al.	
			2010/0270031 A1	10/2010	Patel	
			2010/0304178 A1	12/2010	Dirscherl	
			2011/0048743 A1	3/2011	Stafford et al.	
			2011/0067889 A1	3/2011	Marya et al.	
			2011/0091660 A1	4/2011	Dirscherl	
			2011/0135530 A1	6/2011	Xu et al.	
			2011/0221137 A1	9/2011	Obi et al.	
			2011/0236249 A1	9/2011	Kim et al.	
			2012/0080189 A1	4/2012	Marya et al.	
			2012/0097384 A1	4/2012	Valencia et al.	
			2012/0103135 A1	5/2012	Xu et al.	
			2012/0125642 A1	5/2012	Chenault	
			2012/0156087 A1	6/2012	Kawabata	
			2012/0177905 A1	7/2012	Seals et al.	
			2012/0190593 A1	7/2012	Soane et al.	
			2012/0273229 A1	11/2012	Xu et al.	
			2012/0318513 A1	12/2012	Mazyar et al.	
			2013/0022816 A1	1/2013	Smith et al.	
			2013/0029886 A1	1/2013	Mazyar et al.	
			2013/0032357 A1	2/2013	Mazyar et al.	
			2013/0043041 A1	2/2013	McCoy et al.	
			2013/0047785 A1	2/2013	Xu	
			2013/0056215 A1	3/2013	Crews	
			2013/0068411 A1	3/2013	Forde et al.	
			2013/0112429 A1	5/2013	Crews	
			2013/0133897 A1	5/2013	Bailhly et al.	
			2013/0160992 A1	6/2013	Agrawal et al.	
			2013/0168257 A1	7/2013	Mazyar et al.	
			2013/0199800 A1	8/2013	Kellner et al.	
			2013/0209308 A1	8/2013	Mazyar et al.	
			2013/0220496 A1	8/2013	Inoue et al.	
			2013/0261735 A1	10/2013	Pacetti et al.	
			2013/0048289 A1	12/2013	Mazyar	
			2014/0018489 A1	1/2014	Johnson	
			2014/0027128 A1	1/2014	Johnson	
			2014/0060834 A1	3/2014	Quintero	
			2014/0093417 A1	4/2014	Liu	
			2014/0124216 A1	5/2014	Fripp et al.	
			2014/0190705 A1	7/2014	Fripp	
			2014/0196889 A1	7/2014	Jordan et al.	
			2014/0202284 A1	7/2014	Kim	
			2014/0202708 A1	7/2014	Jacob et al.	
			2014/0219861 A1	8/2014	Han	
			2014/0224477 A1	8/2014	Wiese et al.	
			2014/0236284 A1	8/2014	Stinson	
			2014/0271333 A1	9/2014	Kim et al.	
			2014/0286810 A1	9/2014	Marya	
			2014/0305627 A1	10/2014	Manke	
			2015/0102179 A1	4/2015	McHenry et al.	
			2015/0240337 A1	8/2015	Sherman et al.	
			2015/0247376 A1	9/2015	Tolman	
			2015/0299838 A1	10/2015	Doud	
			2015/0354311 A1	12/2015	Okura et al.	
			2016/0024619 A1	1/2016	Wilks	
			2016/0201425 A1	7/2016	Walton	
			2016/0201427 A1	7/2016	Fripp	
			2016/0201435 A1	7/2016	Fripp et al.	
			2016/0230494 A1	8/2016	Fripp et al.	
			2016/0251934 A1	9/2016	Walton	
			2016/0265091 A1	9/2016	Walton et al.	
	FOREIGN PATENT DOCUMENTS					
2002/0121081 A1	9/2002	Cesaroni et al.	CN	102517489	6/2012	
2002/0197181 A1	12/2002	Osawa et al.	CN	102796928	11/2012	
2003/0173005 A1	9/2003	Higashi	CN	103343271	10/2013	
2005/0194141 A1	9/2005	Sinclair et al.	CN	103602865	2/2014	
2006/0113077 A1	6/2006	Willberg et al.	CN	103898384	7/2014	
2006/0131031 A1	6/2006	McKeachnie	EP	0470599	2/1998	
2006/0175059 A1	8/2006	Sinclair et al.				

## US 10,329,653 B2

Page 3

(56)

## References Cited

## FOREIGN PATENT DOCUMENTS

EP	2088217	8/2009
JP	2008266734	11/2008
JP	2012197491	10/2012
JP	2013019030	1/2013
JP	2014043601	3/2014
KR	20130023707	3/2013
WO	US1990002655	3/1990
WO	1992013978	8/1992
WO	9857347	12/1998
WO	2009055354	4/2009
WO	2012091984	7/2012
WO	2013019410	2/2013
WO	2013019421	2/2013
WO	2013109287	7/2013
WO	2013122712	8/2013
WO	2013154634	10/2013
WO	2014100141	6/2014
WO	2014113058	7/2014
WO	2015171126	11/2015
WO	2016032758	3/2016
WO	2016032761	3/2016
WO	2016036371	3/2016

## OTHER PUBLICATIONS

- Momentive, "Titanium Diborid Powder" condensed product brochure; retrieved from <https://www.momentive.com/WorkArea/DownloadAsset.aspx?id=27489;>; p. 1 (2012).
- Durbin, "Modeling Dissolution in Aluminum Alloys" Dissertation for Georgia Institute of Technology; retrieved from [https://smartech.gatech.edu/bitstream/handle/1853/6873/durbin\\_tracie\\_L\\_200505\\_phd.pdf?](https://smartech.gatech.edu/bitstream/handle/1853/6873/durbin_tracie_L_200505_phd.pdf?) (2005).
- Pegeut et al., "Influence of cold working on the pitting corrosion resistance of stainless steel" Corrosion Science, vol. 49, pp. 1933-1948 (2007).
- Elemental Charts from [chemicalelements.com](http://chemicalelements.com); retrieved Jul. 27, 2017.
- Song et al., "Corrosion Mechanisms of Magnesium Alloys" Advanced Engg Materials, vol. 1, No. 1 (1999).
- Zhou et al., "Tensile Mechanical Properties and Strengthening Mechanism of Hybrid Carbon Nanotubes . . ." Journal of Nanomaterials, 2012; 2012:851862 (doi: 10.1155/2012/851862) Figs. 6 and 7.
- Trojanova et al., "Mechanical and Acoustic Properties of Magnesium Alloys . . ." Light Metal Alloys Application, Chapter 8, Published Jun. 11, 2014 (doi: 10.5772/57454) p. 163, para. [0008], [0014-0015]; [0041-0043].
- AZoNano "Silicon Carbide Nanoparticles—Properties, Applications" <http://www.amazon.com/articles.aspx?ArticleID=3396> p. 2, Physical Properties, Thermal Properties (May 9, 2013).
- AZoM "Magnesium AZ91D-F Alloy" <http://www.amazon.com/articles.aspx?ArticleID=8670> p. 1, Chemical Composition; p. 2 Physical Properties (Jul. 31, 2013).
- Elasser et al., "Silicon Carbide Benefits and Advantages . . ." Proceedings of the IEEE, 2002; 906(6):969-986 (doi: 10.1109/JPROC2002.1021562) p. 970, Table 1.
- Lan et al., "Microstructure and Microhardness of SiC Nanoparticles . . ." Materials Science and Engineering A; 386:284-290 (2004).
- Casati et al., "Metal Matrix Composites Reinforced by Nanoparticles", vol. 4:65-83 (2014).
- Saravanan et al., "Fabrication and characterization of pure magnesium-30 vol. SiCP particle composite", Material Science and Eng., vol. 276, pp. 108-116 (2000).
- Song et al., Texture evolution and mechanical properties of AZ31B magnesium alloy sheets processed by repeated unidirectional bending, Journal of Alloys and Compounds, vol. 489, pp. 475-481 (2010).
- Blawert et al., "Magnesium secondary alloys: Alloy design for magnesium alloys with improved tolerance limits against impurities", Corrosion Science, vol. 52, No. 7, pp. 2452-2468 (Jul. 1, 2010).
- Wang et al., "Effect of Ni on microstructures and mechanical properties of AZ1 02 magnesium alloys" Zhuzao Foundry, Shenyang Zhuzao Yanjiusuo, vol. 62, No. 1, pp. 315-318 (Jan. 1, 2013).
- Kim et al., "Effect of aluminum on the corosions characteristics of Mg—4Ni—xAl alloys", Corrosion, vol. 59, No. 3, pp. 228-237 (Jan. 1, 2003).
- Unsworth et al., "A new magnesium alloy system", Light Metal Age, vol. 37, No. 7-8, pp. 29-32 (Jan. 1, 1979).
- Geng et al., "Enhanced age-hardening response of Mg—Zn alloys via Co additions", Scripta Materialia, vol. 64, No. 6, pp. 506-509 (Mar. 1, 2011).
- Zhu et al., "Microstructure and mechanical properties of Mg6ZnCuO.6Zr (wt.%) alloys", Journal of Alloys and compounds, vol. 509, No. 8, pp. 3526-3531 (Dec. 22, 2010).
- International Search Authority, International Search Report and Written Opinion for PCT/GB2015/052169 (dated Feb. 17, 2016). Search and Examination Report for GB 1413327.6 (dated Jan. 21, 2015).
- Magnesium Elektron Test Report (Mar. 8, 2005).
- New England Fishery Management Counsel, "Fishery Management Plan for American Lobster Amendment 3" (Jul. 1989).
- Emly, E.F., "Principles of Magnesium Technology" Pergamon Press, Oxford (1966).
- Shaw, "Corrosion Resistance of Magnesium Alloys", ASM Handbook, vol. 13A, pp. 692-696 (2003).
- Hanawalt et al., "Corrosion studies of magnesium and its alloys", Metals Technology, Technical Paper 1353 (1941).
- The American Foundry Society, Magnesium alloys, casting source directory 8208, available at [www.afsinc.org/files/magnes.pdf](http://www.afsinc.org/files/magnes.pdf).
- Rokhlin, "Magnesium alloys containing rare earth metals structure and properties", Advances in Metallic Alloys, vol. 3, Taylor & Francis (2003).
- Ghali, "Corrosion Resistance of Aluminum and Magnesium Alloys" pp. 382-389, Wiley Publishing (2010).
- Kim et al., "High Mechanical Strengths of Mg—Ni—Y and Mg—Cu Amorphous Alloys with Significant Supercooled Liquid Region", Materials Transactions, vol. 31, No. 11, pp. 929-934 (1990).
- Tekumalla et al., "Mechanical Properties of Magnesium-Rare Earth Alloy Systems", Metals, vol. 5, pp. 1-39 (2014).
- National Physical Laboratory, "Bimetallic Corrosion" Crown (C) p. 1-14 (2000).

**U.S. Patent**

Jun. 25, 2019

Sheet 1 of 4

**US 10,329,653 B2**

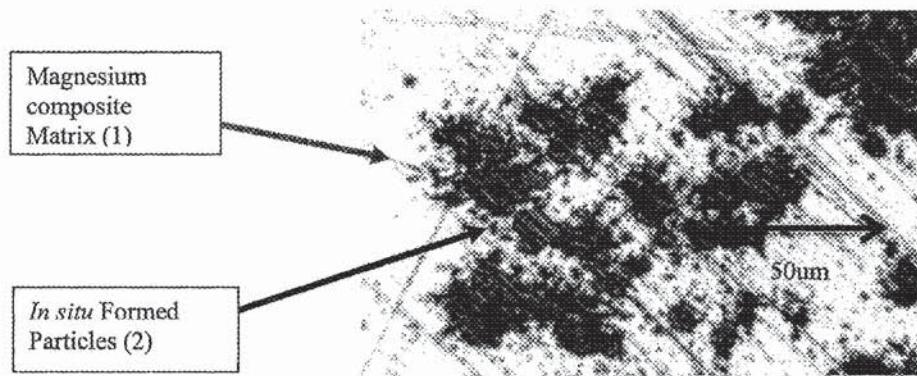


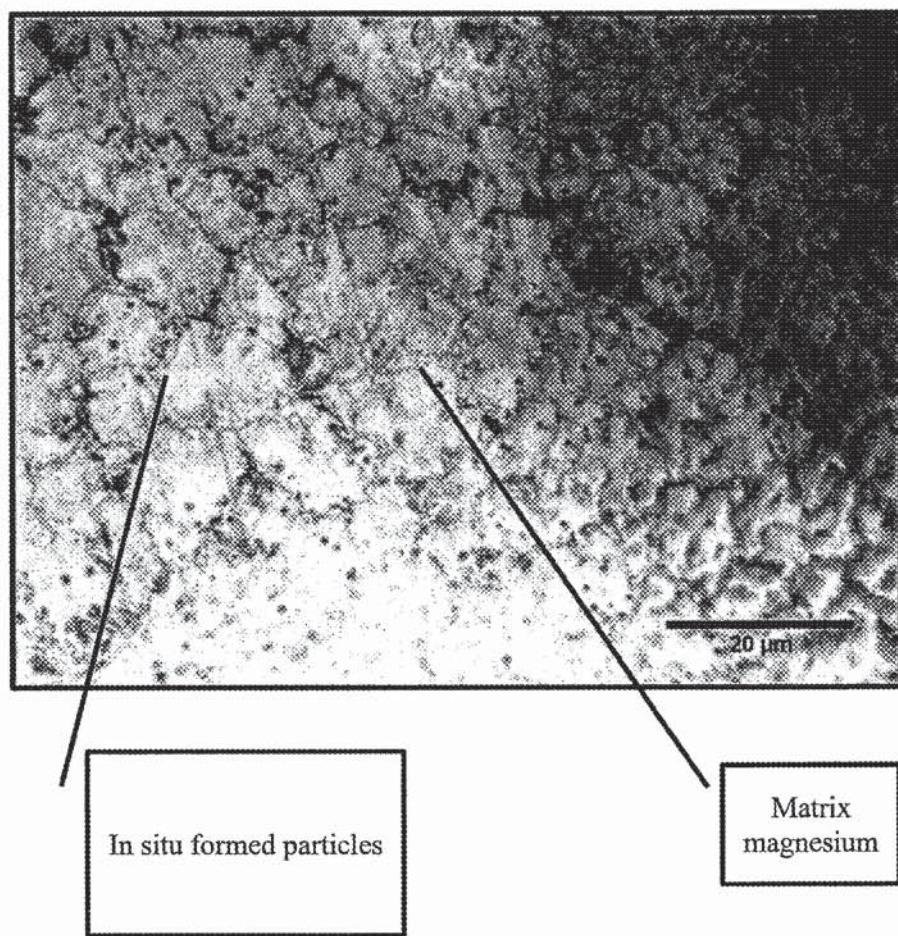
FIG 1.

**U.S. Patent**

Jun. 25, 2019

Sheet 2 of 4

**US 10,329,653 B2**



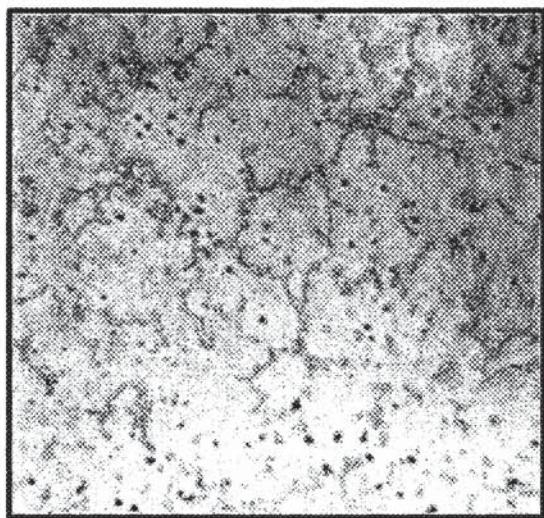
**FIG. 2**

**U.S. Patent**

**Jun. 25, 2019**

**Sheet 3 of 4**

**US 10,329,653 B2**



Highlighted In Situ Formed Particles

**FIG. 3**

U.S. Patent

Jun. 25, 2019

Sheet 4 of 4

US 10,329,653 B2

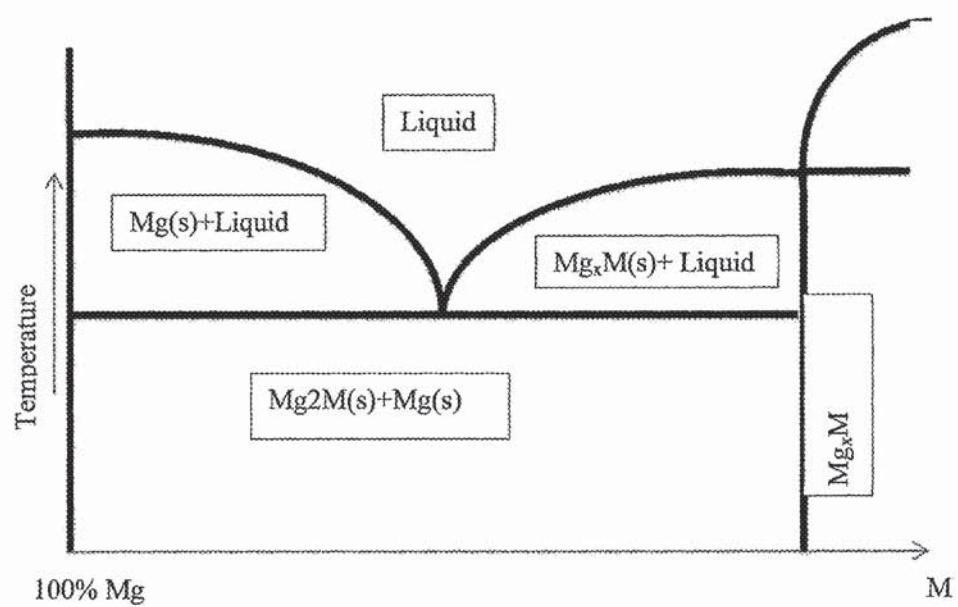


FIG. 4

US 10,329,653 B2

**1**

**GALVANICALLY-ACTIVE IN SITU FORMED  
PARTICLES FOR CONTROLLED RATE  
DISSOLVING TOOLS**

The present invention is a divisional of U.S. patent application Ser. No. 14/689,295 filed Apr. 17, 2015, which in turn claims priority on U.S. Provisional Patent Application Ser. No. 61/981,425 filed Apr. 18, 2014, which is incorporated herein by reference.

**FIELD OF THE INVENTION**

The present invention is directed to a novel magnesium composite for use as a dissolvable component in oil drilling.

**BACKGROUND OF THE INVENTION**

The ability to control the dissolution of a down hole well component in a variety of solutions is very important to the utilization of non-drillable completion tools, such as sleeves, frac balls, hydraulic actuating tooling, and the like. Reactive materials for this application, which dissolve or corrode when exposed to acid, salt, and/or other wellbore conditions, have been proposed for some time. Generally, these components consist of materials that are engineered to dissolve or corrode. Dissolving polymers and some powder metallurgy metals have been disclosed, and are also used extensively in the pharmaceutical industry for controlled release of drugs. Also, some medical devices have been formed of metals or polymers that dissolve in the body.

While the prior art well drill components have enjoyed modest success in reducing well completion costs, their consistency and ability to specifically control dissolution rates in specific solutions, as well as other drawbacks such as limited strength and poor reliability, have impacted their ubiquitous adoption. Ideally, these components would be manufactured by a process that is low cost, scalable, and produces a controlled corrosion rate having similar or increased strength as compared to traditional engineering alloys such as aluminum, magnesium, and iron. Ideally, traditional heat treatments, deformation processing, and machining techniques could be used on the components without impacting the dissolution rate and reliability of such components.

**SUMMARY OF THE INVENTION**

The present invention is directed to a novel magnesium composite for use as a dissolvable component in oil drilling and will be described with particular reference to such application. As can be appreciated, the novel magnesium composite of the present invention can be used in other applications (e.g., non-oil wells, etc.). In one non-limiting embodiment, the present invention is directed to a ball or other tool component in a well drilling or completion operation such as, but not limited to, a component that is seated in a hydraulic operation that can be dissolved away after use so that no drilling or removal of the component is necessary. Tubes, valves, valve components, plugs, frac balls, and other shapes and components can "also be formed of the novel magnesium composite of the present invention. For purposes of this invention, primary dissolution is measured for valve components and plugs as the time the part removes itself from the seat of a valve or plug arrangement or can become free floating in the system. For example, when the part is a plug in a plug system, primary dissolution occurs when the plug has degraded or dissolved to a point

**2**

that it can no longer function as a plug and thereby allows fluid to flow about the plug. For purposes of this invention, secondary dissolution is measured in the time the part is fully dissolved into sub-mm particles. As can be appreciated, the novel magnesium composite of the present invention can be used in other well components that also desire the function of dissolving after a period of time. In one non-limiting aspect of the present invention, a galvanically-active phase is precipitated from the novel magnesium composite composition and is used to control the dissolution rate of the component; however, this is not required. The novel magnesium composite is generally castable and/or machinable, and can be used in place of existing metallic or plastic components in oil and gas drilling rigs including, but not limited to, water injection and hydraulic fracturing. The novel magnesium composite can be heat treated as well as extruded and/or forged.

In one non-limiting aspect of the present invention, the novel magnesium composite is used to form a castable, moldable, or extrudable component. Non-limiting magnesium composites in accordance with the present invention include at least 50 wt % magnesium. One or more additives are added to a magnesium or magnesium alloy to form the novel magnesium composite of the present invention. The one or more additives can be selected and used in quantities so that galvanically-active intermetallic or insoluble precipitates form in the magnesium or magnesium alloy while the magnesium or magnesium alloy is in a molten state and/or during the cooling of the melt; however, this is not required. The one or more additives typically are added in a weight percent that is less than a weight percent of said magnesium or magnesium alloy. Typically, the magnesium or magnesium alloy constitutes about 50.1 wt % 99.9 wt % of the magnesium composite and all values and ranges therebetween. In one non-limiting aspect of the invention, the magnesium or magnesium alloy constitutes about 60 wt. %-95 wt. % of the magnesium composite, and typically the magnesium or magnesium alloy constitutes about 70 wt. %-90 wt. % of the magnesium composite. The one or more additives are typically added to the molten magnesium or magnesium alloy at a temperature that is less than the melting point of the one or more additives. The one or more additives generally have an average particle diameter size of at least about 0.1 microns, typically no more than about 500 microns (e.g., 0.1 microns, 0.1001 microns, 0.1002 microns . . . 499.9998 microns, 499.9999 microns, 500 microns) and including any value or range therebetween, more typically about 0.1 to 400 microns, and still more typically about 10 to 50 microns. During the process of mixing the one or more additives in the molten magnesium or magnesium alloy, the one or more additives are typically not caused to fully melt in the molten magnesium or magnesium alloy. As can be appreciated, the one or more additives can be added to the molten magnesium or magnesium alloy at a temperature that is greater than the melting point of the one or more additives. In such a method of forming the magnesium composite, the one or more additives form secondary metallic alloys with the magnesium and/or other metals in the magnesium alloy, said secondary metallic alloys having a melting point that is greater than the magnesium and/or other metals in the magnesium alloy. As the molten metal cools, these newly formed secondary metallic alloys begin to precipitate out of the molten metal and form in the in situ phase to the matrix phase in the cooled and solid magnesium composite. After the mixing process is completed, the molten magnesium or magnesium alloy and the one or more additives that are mixed in the molten

## US 10,329,653 B2

3

magnesium or magnesium alloy are cooled to form a solid component. Generally, the temperature of the molten magnesium or magnesium alloy is at least about 10° C. less than the melting point of the additive added to the molten magnesium or magnesium alloy during the addition and mixing process, typically at least about 100° C. less than the melting point of the additive added to the molten magnesium or magnesium alloy during the addition and mixing process, more typically about 100° C.-1000° C. (and any value or range therebetween) less than the melting point of the additive added to the molten magnesium or magnesium alloy during the addition and mixing process; however, this is not required. The never melted particles and/or the newly formed secondary metallic alloys are referred to as in situ particle formation in the molten magnesium composite. Such a process can be used to achieve a specific galvanic corrosion rate in the entire magnesium composite and/or along the grain boundaries of the magnesium composite.

The invention adopts a feature that is usually a negative in traditional casting practices wherein a particle is formed during the melt processing that corrodes the alloy when exposed to conductive fluids and is imbedded in eutectic phases, the grain boundaries, and/or even within grains with precipitation hardening. This feature results in the ability to control where the galvanically-active phases are located in the final casting, as well as the surface area ratio of the in situ phase to the matrix phase, which enables the use of lower cathode phase loadings as compared to a powder metallurgical or alloyed composite to achieve the same dissolution rates. The in situ formed galvanic additives can be used to enhance mechanical properties of the magnesium composite such as ductility, tensile strength, and/or shear strength. The final magnesium composite can also be enhanced by heat treatment as well as deformation processing (such as extrusion, forging, or rolling) to further improve the strength of the final composite over the as-cast material; however, this is not required. The deformation processing can be used to achieve strengthening of the magnesium composite by reducing the grain size of the magnesium composite. Further enhancements, such as traditional alloy heat treatments (such as solutionizing, aging and/or cold working) can be used to enable control of dissolution rates though precipitation of more or less galvanically-active phases within the alloy microstructure while improving mechanical properties; however, this is not required. Because galvanic corrosion is driven by both the electro potential between the anode and cathode phase, as well as the exposed surface area of the two phases, the rate of corrosion can also be controlled through adjustment of the in situ formed particles size, while not increasing or decreasing the volume or weight fraction of the addition, and/or by changing the volume/weight fraction without changing the particle size. Achievement of in situ particle size control can be achieved by mechanical agitation of the melt, ultrasonic processing of the melt, controlling cooling rates, and/or by performing heat treatments. In situ particle size can also or alternatively be modified by secondary processing such as rolling, forging, extrusion and/or other deformation techniques.

In another non-limiting aspect of the invention, a cast structure can be made into almost any shape. During formation, the active galvanically-active in situ phases can be uniformly dispersed throughout the component and the grain or the grain boundary composition can be modified to achieve the desired dissolution rate. The galvanic corrosion can be engineered to affect only the grain boundaries and/or can affect the grains as well (based on composition); however, this is not required. This feature can be used to enable

4

fast dissolutions of high-strength lightweight alloy composites with significantly less active (cathode) in situ phases as compared to other processes.

In still another and/or alternative non-limiting aspect of the invention, ultrasonic processing can be used to control the size of the in situ formed galvanically-active phases; however, this is not required.

In yet another and/or alternative non-limiting aspect of the invention, the in situ formed particles can act as matrix strengtheners to further increase the tensile strength of the material compared to the base alloy without the additive; however, this is not required.

In still yet another and/or alternative non-limiting aspect of the invention, there is provided a method of controlling the dissolution properties of a metal selected from the class of magnesium and/or magnesium alloy comprising of the steps of a) melting the magnesium or magnesium alloy to a point above its solidus, b) introducing an additive material and/or phase to the magnesium or magnesium alloy in order to achieve in situ precipitation of galvanically-active intermetallic phases, and c) cooling the melt to a solid form. The additive material is generally added to the magnesium or magnesium alloy when the magnesium or magnesium alloy is in a molten state and at a temperature that is less than the melting point of the additive material. The galvanically-active intermetallic phases can be used to enhance the yield strength of the alloy; however, this is not required. The size of the in situ precipitated intermetallic phase can be controlled by a melt mixing technique and/or cooling rate; however, this is not required. The method can include the additional step of subjecting the magnesium composite to intermetallic precipitates to solutionizing of at least about 300° C. to improve tensile strength and/or improve ductility; however, this is not required. The solutionizing temperature is less than the melting point of the magnesium composite. Generally, the solutionizing temperature is less than 50° C.-200° C. (the melting point of the magnesium composite) and the time period of solutionizing is at least 0.1 hours. In one non-limiting aspect of the invention, the magnesium composite can be subjected to a solutionizing temperature for about 0.5-50 hours (e.g., 1-15 hours, etc.) at a temperature of 300° C.-620° C. (e.g., 300° C.-500° C., etc.). The method can include the additional step of subjecting the magnesium composite to intermetallic precipitates and to artificially age the magnesium composite at a temperature at least about 90° C. to improve the tensile strength; however, this is not required. The artificially aging process temperature is typically less than the solutionizing temperature and the time period of the artificially aging process temperature is typically at least 0.1 hours. Generally, the artificially aging process is less than 50° C.-400° C. (the solutionizing temperature). In one non-limiting aspect of the invention, the magnesium composite can be subjected to aging treatment for about 0.5-50 hours (e.g., 1-16 hours, etc.) at a temperature of 90° C.-300° C. (e.g., 100° C.-200° C.).

In another and/or alternative non-limiting aspect of the invention, there is provided a magnesium composite that is over 50 wt. % magnesium and about 0.05-35 wt. % nickel (and all values or ranges therebetween) is added to the magnesium or magnesium alloy to form intermetallic Mg<sub>2</sub>Ni as a galvanically-active in situ precipitate. In one non-limiting arrangement, the magnesium composite includes about 0.05-23.5 wt. % nickel, 0.01-5 wt. % nickel, 3-7 wt. % nickel, 7-10 wt. % nickel, or 10-24.5 wt. % nickel. The nickel is added to the magnesium or magnesium alloy while the temperature of the molten magnesium or magnesium alloy is less than the melting point of the nickel. Throughout

## US 10,329,653 B2

5

the mixing process, the temperature of the molten magnesium or magnesium alloy is less than the melting point of the nickel. During the mixing process, solid particles of  $Mg_2Ni$  are formed. Once the mixing process is complete, the mixture of molten magnesium or magnesium alloy, solid particles of  $Mg_2Ni$ , and any unalloyed nickel particles are cooled and an in situ precipitate of solid particles of  $Mg_2Ni$  and any unalloyed nickel particles are formed in the solid magnesium or magnesium alloy. Generally, the temperature of the molten magnesium or magnesium alloy is at least about 200° C. less than the melting point of the nickel added to the molten magnesium or magnesium alloy during the addition and mixing process.

In still another and/or alternative non-limiting aspect of the invention, there is provided a magnesium composite that is over 50 wt. % magnesium and about 0.05-35 wt. % copper (and all values or ranges therebetween) is added to the magnesium or magnesium alloy to form intermetallic  $CuMg_2$  as the galvanically-active in situ precipitate. In one non-limiting arrangement, the magnesium composite includes about 0.01-5 wt. % copper, about 0.5-15 wt. % copper, about 15-35 wt. % copper, or about 0.01-20 wt. %. The copper is added to the magnesium or magnesium alloy while the temperature of the molten magnesium or magnesium alloy is less than the melting point of the copper. Throughout the mixing process, the temperature of the molten magnesium or magnesium alloy is less than the melting point of the copper. During the mixing process, solid particles of  $CuMg_2$  are formed. Once the mixing process is complete, the mixture of molten magnesium or magnesium alloy, solid particles of  $CuMg_2$ , and any unalloyed copper particles are cooled and an in situ precipitate of solid particles of  $CuMg_2$  and any unalloyed copper particles are formed in the solid magnesium or magnesium alloy. Generally, the temperature of the molten magnesium or magnesium alloy is at least about 200° C. less than the melting point of the copper added to the molten magnesium or magnesium alloy.

In yet another and/or alternative non-limiting aspect of the invention, there is provided a magnesium composite that is over 50 wt. % magnesium and about 0.05-20% by weight cobalt is added to the magnesium or magnesium alloy to form an intermetallic  $CoMg_2$  as the galvanically-active in situ precipitate. The cobalt is added to the magnesium or magnesium alloy while the temperature of the molten magnesium or magnesium alloy is less than the melting point of the cobalt. Throughout the mixing process, the temperature of the molten magnesium or magnesium alloy is less than the melting point of the cobalt. During the mixing process, solid particles of  $CoMg_2$  are formed. Once the mixing process is complete, the mixture of molten magnesium or magnesium alloy, solid particles of  $CoMg_2$ , and any unalloyed cobalt particles are cooled and an in situ precipitate of solid particles of  $CoMg_2$  and any unalloyed cobalt particles are formed in the solid magnesium or magnesium alloy. Generally, the temperature of the molten magnesium or magnesium alloy is at least about 200° C. less than the melting point of the cobalt added to the molten magnesium or magnesium alloy.

In yet another and/or alternative non-limiting aspect of the invention, there is provided a magnesium composite that is over 50 wt. % magnesium and cobalt is added to the magnesium or magnesium alloy which forms an intermetallic  $Mg_xCo$  as the galvanically-active particle in situ precipitate. The cobalt is added to the magnesium or magnesium alloy while the temperature of the molten magnesium or magnesium alloy is less than the melting point of the

6

cobalt. Throughout the mixing process, the temperature of the molten magnesium or magnesium alloy is less than the melting point of the cobalt. During the mixing process, solid particles of  $CoMg_x$  are formed. Once the mixing process is complete, the mixture of molten magnesium or magnesium alloy, solid particles of  $CoMg_x$ , and any unalloyed cobalt particles are cooled and an in situ precipitate of solid particles of  $CoMg_x$  and any unalloyed cobalt particles are formed in the solid magnesium or magnesium alloy. Generally, the temperature of the molten magnesium or magnesium alloy is at least about 200° C. less than the melting point of the cobalt added to the molten magnesium or magnesium alloy.

In still yet another and/or alternative non-limiting aspect of the invention, there is provided a magnesium composite that is over 50 wt. % magnesium and about 0.5-35% by weight of secondary metal (SM) is added to the magnesium or magnesium alloy to form a galvanically active intermetallic particle when compared to magnesium or a magnesium alloy in the remaining casting where the cooling rate between the liquidus to the solidus is faster than 1° C. per minute. The secondary metal is added to the magnesium or magnesium alloy while the temperature of the molten magnesium or magnesium alloy is less than the melting point of the secondary metal. Throughout the mixing process, the temperature of the molten magnesium or magnesium alloy is less than the melting point of the secondary metal. During the mixing process, solid particles of  $SMMg_x$  are formed. Once the mixing process is complete, the mixture of molten magnesium or magnesium alloy, solid particles of  $SMMg_x$ , and any unalloyed secondary metal particles are cooled and an in situ precipitate of solid particles of  $SMMg_x$  and any unalloyed secondary metal particles are formed in the solid magnesium or magnesium alloy. Generally, the temperature of the molten magnesium or magnesium alloy is at least about 200° C. less than the melting point of the secondary metal added to the molten magnesium or magnesium alloy. As can be appreciated, one or more secondary metals can be added to the molten magnesium or magnesium alloy.

In another and/or alternative non-limiting aspect of the invention, there is provided a magnesium composite that is over 50 wt. % magnesium and about 0.5-35% by weight of secondary metal (SM) is added to the magnesium or magnesium alloy to form a galvanically active intermetallic particle when compared to magnesium or a magnesium alloy in the remaining casting where the cooling rate between the liquidus to the solidus is slower than 1° C. per minute. The secondary metal is added to the magnesium or magnesium alloy while the temperature of the molten magnesium or magnesium alloy is less than the melting point of the secondary metal. Throughout the mixing process, the temperature of the molten magnesium or magnesium alloy is less than the melting point of the secondary metal. During the mixing process, solid particles of  $SMMg_x$  are formed. Once the mixing process is complete, the mixture of molten magnesium or magnesium alloy, solid particles of  $SMMg_x$ , and any unalloyed secondary metal particles are cooled and an in situ precipitate of solid particles of  $SMMg_x$  and any unalloyed secondary metal particles are formed in the solid magnesium or magnesium alloy. Generally, the temperature of the molten magnesium or magnesium alloy is at least about 200° C. less than the melting point of the secondary metal added to the molten magnesium or magnesium alloy. As can be appreciated, one or more secondary metals can be added to the molten magnesium or magnesium alloy.

In still another and/or alternative non-limiting aspect of the invention, there is provided a magnesium composite that

## US 10,329,653 B2

7

is over 50 wt. % magnesium and about 0.05-35 wt. % of secondary metal (SM) is added to the magnesium or magnesium alloy to form a galvanically-active intermetallic particle when compared to magnesium or a magnesium alloy in the remaining casting where the cooling rate between the liquidus to the solidus is faster than 0.01° C. per min and slower than 1° C. per minute. The secondary metal is added to the magnesium or magnesium alloy while the temperature of the molten magnesium or magnesium alloy is less than the melting point of the secondary metal. Throughout the mixing process, the temperature of the molten magnesium or magnesium alloy is less than the melting point of the secondary metal. During the mixing process, solid particles of SMMg<sub>x</sub> are formed. Once the mixing process is complete, the mixture of molten magnesium or magnesium alloy, solid particles of SMMg<sub>x</sub> and any unalloyed secondary metal particles are cooled and an in situ precipitate of solid particles of SMMg<sub>x</sub> and any unalloyed secondary metal particles are formed in the solid magnesium or magnesium alloy. Generally, the temperature of the molten magnesium or magnesium alloy is at least about 200° C. less than the melting point of the secondary metal added to the molten magnesium or magnesium alloy. As can be appreciated, one or more secondary metals can be added to the molten magnesium or magnesium alloy.

In yet another and/or alternative non-limiting aspect of the invention, there is provided a magnesium composite that is over 50 wt. % magnesium and about 0.05-35 wt. % of secondary metal (SM) is added to the magnesium or magnesium alloy to form a galvanically-active intermetallic particle when compared to magnesium or a magnesium alloy in the remaining casting where the cooling rate between the liquidus to the solidus is faster than 10° C. per minute. The secondary metal is added to the magnesium or magnesium alloy while the temperature of the molten magnesium or magnesium alloy is less than the melting point of the secondary metal. Throughout the mixing process, the temperature of the molten magnesium or magnesium alloy is less than the melting point of the secondary metal. During the mixing process, solid particles of SMMg<sub>x</sub> were formed. Once the mixing process was completed, the mixture of molten magnesium or magnesium alloy, solid particles of SMMg<sub>x</sub>, and any unalloyed secondary metal particles are cooled and an in situ precipitate of solid particles of SMMg<sub>x</sub> and any unalloyed secondary metal particles are formed in the solid magnesium or magnesium alloy. Generally, the temperature of the molten magnesium or magnesium alloy is at least about 200° C. less than the melting point of the secondary metal added to the molten magnesium or magnesium alloy. As can be appreciated, one or more secondary metals can be added to the molten magnesium or magnesium alloy.

In still yet another and/or alternative non-limiting aspect of the invention, there is provided magnesium composite that is over 50 wt. % magnesium and about 0.5-35 wt. % of secondary metal (SM) is added to the magnesium or magnesium alloy to form a galvanically-active intermetallic particle when compared to magnesium or a magnesium alloy in the remaining casting where the cooling rate between the liquidus to the solidus is slower than 10° C. per minute. The secondary metal is added to the magnesium or magnesium alloy while the temperature of the molten magnesium or magnesium alloy is less than the melting point of the secondary metal. Throughout the mixing process, the temperature of the molten magnesium or magnesium alloy is less than the melting point of the secondary metal. During the mixing process, solid particles of SMMg<sub>x</sub> are formed.

8

Once the mixing process is complete, the mixture of molten magnesium or magnesium alloy, solid particles of SMMg<sub>x</sub>, and any unalloyed secondary metal particles are cooled and an in situ precipitate of solid particles of SMMg<sub>x</sub> and any unalloyed secondary metal particles are formed in the solid magnesium or magnesium alloy. Generally, the temperature of the molten magnesium or magnesium alloy is at least about 200° C. less than the melting point of the secondary metal added to the molten magnesium or magnesium alloy.

10 As can be appreciated, one or more secondary metals can be added to the molten magnesium or magnesium alloy.

In another and/or alternative non-limiting aspect of the invention, there is provided a magnesium alloy that includes over 50 wt. % magnesium and includes at least one metal selected from the group consisting of aluminum in an amount of about 0.5-10 wt. %, zinc in amount of about 0.05-6 wt. %, zirconium in an amount of about 0.01-3 wt. %, and/or manganese in an amount of about 0.15-2 wt. %. In one non-limiting formulation, the magnesium alloy that includes over 50 wt. % magnesium and includes at least one metal selected from the group consisting of zinc in amount of about 0.05-6 wt. %, zirconium in an amount of about 0.05-3 wt. %, manganese in an amount of about 0.05-0.25 wt. %, boron in an amount of about 0.0002-0.04 wt. %, and bismuth in an amount of about 0.4-0.7 wt. %. The magnesium alloy can then be heated to a molten state and one or more secondary metal (SM) (e.g., copper, nickel, cobalt, titanium, silicon, iron, etc.) can be added to the molten magnesium alloy which forms an intermetallic galvanically-active particle in situ precipitate. The galvanically-active particle can be SMMg<sub>x</sub>, SMAI<sub>x</sub>, SMZn<sub>x</sub>, SMZr<sub>x</sub>, SMMn<sub>x</sub>, SMB<sub>x</sub>, SMBi<sub>x</sub>, SM in combination with anyone of B, Bi, Mg, Al, Zn, Zr, and Mn.

In still another and/or alternative non-limiting aspect of the invention, there is provided a magnesium composite that is over 50 wt. % magnesium and at least one metal selected from the group consisting of zinc in an amount of about 0.05-6 wt. %, zirconium in amount of about 0.05-3 wt. %, manganese in an amount of about 0.05-0.25 wt. %, boron in an amount of about 0.0002-0.04 wt. %, and/or bismuth in an amount of about 0.4-0.7 wt. % is added to the magnesium or magnesium alloy to form a galvanically-active intermetallic particle in the magnesium or magnesium alloy. The magnesium alloy can then be heated to a molten state and one or more secondary metal (SM) (e.g., copper, nickel, cobalt, titanium, iron, etc.) can be added to the molten magnesium alloy which forms an intermetallic galvanically-active particle in situ precipitate. The galvanically-active particle can be SMMg<sub>x</sub>, SMZn<sub>x</sub>, SMZr<sub>x</sub>, SMMn<sub>x</sub>, SMB<sub>x</sub>, SMBi<sub>x</sub>, SM in combination with anyone of Mg, Zn, Zr, Mn, B and/or Bi.

In yet another and/or alternative non-limiting aspect of the invention, there is provided a magnesium or magnesium alloy that is over 50 wt. % magnesium and nickel in an amount of about 0.01-5 wt. % is added to the magnesium or magnesium alloy to form a galvanically-active intermetallic particle in the magnesium or magnesium alloy. The nickel is added to the magnesium or magnesium alloy while the temperature of the molten magnesium or magnesium alloy is less than the melting point of the nickel. Throughout the mixing process, the temperature of the molten magnesium or magnesium alloy is less than the melting point of the nickel. During the mixing process, solid particles of Mg<sub>2</sub>Ni are formed. Once the mixing process is complete, the mixture of molten magnesium or magnesium alloy, solid particles of Mg<sub>2</sub>Ni, and any unalloyed nickel particles are cooled and an in situ precipitate of solid particles of Mg<sub>2</sub>Ni and any unalloyed nickel particles are formed in the solid magne-



## US 10,329,653 B2

**11**

C. less than the melting point of the copper added to the molten magnesium or magnesium alloy during the addition and mixing process.

In still another and/or alternative non-limiting aspect of the invention, there is provided a magnesium composite that is over 50 wt. % magnesium and includes copper in an amount of about 0.01-20 wt. % is added to the magnesium or magnesium alloy to form a galvanically-active intermetallic particle in the magnesium or magnesium alloy. The copper is added to the magnesium or magnesium alloy while the temperature of the molten magnesium or magnesium alloy is less than the melting point of the copper. Throughout the mixing process, the temperature of the molten magnesium or magnesium alloy is less than the melting point of the copper. During the mixing process, solid particles of  $Mg_2Cu$  are formed. Once the mixing process is complete, the mixture of molten magnesium or magnesium alloy, solid particles of  $Mg_2Cu$ , and any unalloyed nickel particles are cooled and an in situ precipitate of solid particles of  $Mg_2Cu$  and any unalloyed copper particles are formed in the solid magnesium or magnesium alloy. Generally, the temperature of the molten magnesium or magnesium alloy is at least about 200° C. less than the melting point of the copper added to the molten magnesium or magnesium alloy during the addition and mixing process.

In yet another and/or alternative non-limiting aspect of the invention, there is provided a magnesium composite that is subjected to heat treatments such as solutionizing, aging and/or cold working to be used to control dissolution rates though precipitation of more or less galvanically-active phases within the alloy microstructure while improving mechanical properties. The aging process (when used) can be for at least about 1 hour, for about 1-50 hours, for about 1-20 hours, or for about 8-20 hours. The solutionizing (when used) can be for at least about 1 hour, for about 1-50 hours, for about 1-20 hours, or for about 8-20 hours.

In still yet another and/or alternative non-limiting aspect of the invention, there is provided a method for controlling the dissolution rate of the magnesium composite wherein the magnesium content is at least about 75% and nickel is added to form in situ precipitation of at least 0.05 wt. %  $MgNi_2$  with the magnesium or magnesium alloy and solutionizing the resultant metal at a temperature within a range of 100-500° C. for a period of 0.25-50 hours, the magnesium composite being characterized by higher dissolution rates than metal without nickel additions subjected to the said aging treatment.

In another and/or alternative non-limiting aspect of the invention, there is provided a method for improving the physical properties of the magnesium composite wherein the magnesium content is at least about 85% and nickel is added to form in situ precipitation of at least 0.05 wt. %  $MgNi_2$  with the magnesium or magnesium alloy and solutionizing the resultant metal at a temperature at about 100-500° C. for a period of 0.25-50 hours, the magnesium composite being characterized by higher tensile and yield strengths than magnesium base alloys of the same composition, but not including the amount of nickel.

In still another and/or alternative non-limiting aspect of the invention, there is provided a method for controlling the dissolution rate of the magnesium composite wherein the magnesium content in the alloy is at least about 75% and copper is added to form in situ precipitation of at least about 0.05 wt. %  $MgCu_2$  with the magnesium or magnesium alloy and solutionizing the resultant metal at a temperature within a range of 100-500° C. for a period of 0.25-50 hours, the

**12**

magnesium composite being characterized by higher dissolution rates than metal without copper additions subjected to the said aging treatment.

In yet another and/or alternative non-limiting aspect of the invention, there is provided a method for improving the physical properties of the magnesium composite wherein the total content of magnesium in the magnesium or magnesium alloy is at least about 85 wt. % and copper is added to form in situ precipitation of at least 0.05 wt. %  $MgCu_2$  with the magnesium or magnesium composite and solutionizing the resultant metal at a temperature of about 100-500° C. for a period of 0.25-50 hours, the magnesium composite is characterized by higher tensile and yield strengths than magnesium base alloys of the same composition, but not including the amount of copper.

In still yet another and/or alternative non-limiting aspect of the invention, there is provided a magnesium composite for use as a dissolvable ball or frac ball in hydraulic fracturing and well drilling.

In another and/or alternative non-limiting aspect of the invention, there is provided a magnesium composite for use as a dissolvable tool for use in well drilling and hydraulic control as well as hydraulic fracturing.

In still another and/or alternative non-limiting aspect of the invention, there is provided a magnesium composite that includes secondary institute formed reinforcements that are not galvanically-active to the magnesium or magnesium alloy matrix to increase the mechanical properties of the magnesium composite. The secondary institute formed reinforcements include a  $Mg_2Si$  phase as the in situ formed reinforcement.

In yet another and/or alternative non-limiting aspect of the invention, there is provided a magnesium composite that is subjected to a faster cooling rate from the liquidus to the solidus point to create smaller in situ formed particles.

In still yet another and/or alternative non-limiting aspect of the invention, there is provided a magnesium composite that is subjected to a slower cooling rate from the liquidus to the solidus point to create larger in situ formed particles.

In yet another and/or alternative non-limiting aspect of the invention, there is provided a magnesium composite that is subjected to heat treatments such as solutionizing, aging and/or cold working to be used to control dissolution rates though precipitation of more or less galvanically-active phases within the alloy microstructure while improving mechanical properties. The aging process (when used) can be for at least about 1 hour, for about 1-50 hours, for about 1-20 hours, or for about 8-20 hours. The solutionizing (when used) can be for at least about 1 hour, for about 1-50 hours, for about 1-20 hours, or for about 8-20 hours.

In still yet another and/or alternative non-limiting aspect of the invention, there is provided a method for controlling the dissolution rate of the magnesium composite wherein the magnesium content is at least about 75% and nickel is added to form in situ precipitation of at least 0.05 wt. %  $MgNi_2$  with the magnesium or magnesium alloy and solutionizing the resultant metal at a temperature within a range of 100-500° C. for a period of 0.25-50 hours, the magnesium composite being characterized by higher dissolution rates than metal without nickel additions subjected to the said aging treatment.

In another and/or alternative non-limiting aspect of the invention, there is provided a method for improving the physical properties of the magnesium composite wherein the magnesium content is at least about 85% and nickel is added to form in situ precipitation of at least 0.05 wt. %  $MgNi_2$  with the magnesium or magnesium alloy and solutionizing

## US 10,329,653 B2

13

the resultant metal at a temperature at about 100-500° C. for a period of 0.25-50 hours, the magnesium composite being characterized by higher tensile and yield strengths than magnesium base alloys of the same composition, but not including the amount of nickel.

In still another and/or alternative non-limiting aspect of the invention, there is provided a method for controlling the dissolution rate of the magnesium composite wherein the magnesium content in the alloy is at least about 75% and copper is added to form in situ precipitation of at least about 0.05 wt. % MgCu<sub>2</sub> with the magnesium or magnesium alloy and solutionizing the resultant metal at a temperature within a range of 100-500° C. for a period of 0.25-50 hours, the magnesium composite being characterized by higher dissolution rates than metal without copper additions subjected to the said aging treatment.

In yet another and/or alternative non-limiting aspect of the invention, there is provided a method for improving the physical properties of the magnesium composite wherein the total content of magnesium in the magnesium or magnesium alloy is at least about 85 wt. % and copper is added to form in situ precipitation of at least 0.05 wt. % MgCu<sub>2</sub> with the magnesium or magnesium composite and solutionizing the resultant metal at a temperature of about 100-500° C. for a period of 0.25-50 hours, the magnesium composite is characterized by higher tensile and yield strengths than magnesium base alloys of the same composition, but not including the amount of copper.

In still yet another and/or alternative non-limiting aspect of the invention, there is provided a magnesium composite for use as a dissolvable ball or frac ball in hydraulic fracturing and well drilling.

In another and/or alternative non-limiting aspect of the invention, there is provided a magnesium composite for use as a dissolvable tool for use in well drilling and hydraulic control as well as hydraulic fracturing.

In still another and/or alternative non-limiting aspect of the invention, there is provided a magnesium composite that includes secondary institute formed reinforcements that are not galvanically-active to the magnesium or magnesium alloy matrix to increase the mechanical properties of the magnesium composite. The secondary institute formed reinforcements include a Mg<sub>2</sub>Si phase as the in situ formed reinforcement.

In yet another and/or alternative non-limiting aspect of the invention, there is provided a magnesium composite that is subjected to a faster cooling rate from the liquidus to the solidus point to create smaller in situ formed particles.

In still yet another and/or alternative non-limiting aspect of the invention, there is provided a magnesium composite that is subjected to a slower cooling rate from the liquidus to the solidus point to create larger in situ formed particles.

In another and/or alternative non-limiting aspect of the invention, there is provided a magnesium composite that is subjected to mechanical agitation during the cooling rate from the liquidus to the solidus point to create smaller in situ formed particles.

In another and/or alternative non-limiting aspect of the invention, there is provided a magnesium composite that is subjected to mechanical agitation during the cooling rate from the liquidus to the solidus point to create smaller in situ formed particles.

In still another and/or alternative non-limiting aspect of the invention, there is provided a magnesium composite that is subjected to chemical agitation during the cooling rate from the liquidus to the solidus point to create smaller in situ formed particles.

14

In yet another and/or alternative non-limiting aspect of the invention, there is provided a magnesium composite that is subjected to ultrasonic agitation during the cooling rate from the liquidus to the solidus point to create smaller in situ formed particles.

In still yet another and/or alternative non-limiting aspect of the invention, there is provided a magnesium composite that is subjected to deformation or extrusion to further improve dispersion of the in situ formed particles.

In another and/or alternative non-limiting aspect of the invention, there is provided a method for forming a novel magnesium composite including the steps of a) selecting an AZ91D magnesium alloy having 9 wt. % aluminum, 1 wt. % zinc and 90 wt. % magnesium, b) melting the AZ91 D magnesium alloy to a temperature above 800° C., c) adding up to about 7 wt. % nickel to the melted AZ91D magnesium alloy at a temperature that is less than the melting point of nickel, d) mixing the nickel with the melted AZ91D magnesium alloy and dispersing the nickel in the melted alloy using chemical mixing agents while maintaining the temperature below the melting point of nickel, and e) cooling and casting the melted mixture in a steel mold. The cast material has a tensile strength of about 14 ksi, and an elongation of about 3% and a shear strength of 11 ksi. The cast material has a dissolve rate of about 75 mg/cm<sup>2</sup>-min in a 3% KCl solution at 90° C. The cast material dissolves at a rate of 1 mg/cm<sup>2</sup>-hr in a 3% KCl solution at 21° C. The cast material dissolves at a rate of 325 mg/cm<sup>2</sup>-hr. in a 3% KCl solution at 90° C. The cast material can be subjected to extrusion with a 11:1 reduction area. The extruded cast material exhibits a tensile strength of 40 ksi, and an elongation to failure of 12%. The extruded cast material dissolves at a rate of 0.8 mg/cm<sup>2</sup>-min in a 3% KCl solution at 20° C. The extruded cast material dissolves at a rate of 100 mg/cm<sup>2</sup>-hr. in a 3% KCl solution at 90° C. The extruded cast material can be subjected to an artificial T5 age treatment of 16 hours between 100° C.-200° C. The aged extruded cast material exhibits a tensile strength of 48 ksi, an elongation to failure of 5%, and a shear strength of 25 ksi. The aged extruded cast material dissolves at a rate of 110 mg/cm<sup>2</sup>-hr in 3% KCl solution at 90° C. and 1 mg/cm<sup>2</sup>-hr in 3% KCl solution at 20° C. The cast material can be subjected to a solutionizing treatment T4 for about 18 hours between 400° C.-500° C. and then subjected to an artificial T6 age treatment for about 16 hours between 100° C.-200° C. The aged and solutionized cast material exhibits a tensile strength of about 34 ksi, an elongation to failure of about 11%, and a shear strength of about 18 ksi. The aged and solutionized cast material dissolves at a rate of about 84 mg/cm<sup>2</sup>-hr in 3% KCl solution at 90° C., and about 0.8 mg/cm<sup>2</sup>-hr in 3% KCl solution at 20° C.

In another and/or alternative non-limiting aspect of the invention, there is provided a method for forming a novel magnesium composite including the steps of a) selecting an AZ91D magnesium alloy having 9 wt. % aluminum, 1 wt. % zinc and 90 wt. % magnesium, b) melting the AZ91D magnesium alloy to a temperature above 800° C., c) adding up to about 1 wt. % nickel to the melted AZ91D magnesium alloy at a temperature that is less than the melting point of nickel, d) mixing the nickel with the melted AZ91D magnesium alloy and dispersing the nickel in the melted alloy using chemical mixing agents while maintaining the temperature below the melting point of nickel, and e) cooling and casting the melted mixture in a steel mold. The cast material has a tensile strength of about 18 ksi, and an elongation of about 5% and a shear strength of 17 ksi. The cast material has a dissolve rate of about 45 mg/cm<sup>2</sup>-min in

US 10,329,653 B2

**15**

a 3% KCl solution at 90° C. The cast material dissolves at a rate of 0.5 mg/cm<sup>2</sup>-hr. in a 3% KCl solution at 21° C. The cast material dissolves at a rate of 325 mg/cm<sup>2</sup>-hr. in a 3% KCl solution at 90° C. The cast material was then subjected to extrusion with a 20:1 reduction area. The extruded cast material exhibits a tensile yield strength of 35 ksi, and an elongation to failure of 12%. The extruded cast material dissolves at a rate of 0.8 mg/cm<sup>2</sup>-min in a 3% KCl solution at 20° C. The extruded cast material dissolves at a rate of 50 mg/cm<sup>2</sup>-hr in a 3% KCl solution at 90° C. The extruded cast material can be subjected to an artificial T5 age treatment of 16 hours between 100° C.-200° C. The aged extruded cast material exhibits a tensile strength of 48 ksi, an elongation to failure of 5%, and a shear strength of 25 ksi.

In still another and/or alternative non-limiting aspect of the invention, there is provided a method for forming a novel magnesium composite including the steps of a) selecting an AZ91D magnesium alloy having about 9 wt. % aluminum, 1 wt. % zinc and 90 wt. % magnesium, b) melting the AZ91D magnesium alloy to a temperature above 800° C., c) adding about 10 wt. % copper to the melted AZ91D magnesium alloy at a temperature that is less than the melting point of copper, d) dispersing the copper in the melted AZ91D magnesium alloy using chemical mixing agents at a temperature that is less than the melting point of copper, and e) cooling casting the melted mixture in a steel mold. The cast material exhibits a tensile strength of about 14 ksi, an elongation of about 3%, and shear strength of 11 ksi. The cast material dissolves at a rate of about 50 mg/cm<sup>2</sup>-hr. in a 3% KCl solution at 90° C. The cast material dissolves at a rate of 0.6 mg/cm<sup>2</sup>-hr. in a 3% KCl solution at 21° C. The cast material can be subjected to an artificial T5 age treatment for about 16 hours at a temperature of 100-200° C. The aged cast material exhibits a tensile strength of 50 Ksi, an elongation to failure of 5%, and a shear strength of 25 ksi. The aged cast material dissolved at a rate of 40 mg/cm<sup>2</sup>-hr in 3% KCl solution at 90° C. and 0.5 mg/cm<sup>2</sup>-hr in 3% KCl solution at 20° C.

These and other objects, features and advantages of the present invention will become apparent in light of the following detailed description of preferred embodiments thereof, as illustrated in the accompanying drawings.

**BRIEF DESCRIPTION OF THE DRAWINGS**

FIGS. 1-3 show a typical cast microstructure with galvanically-active in situ formed intermetallic phase wetted to the magnesium matrix; and,

FIG. 4 shows a typical phase diagram to create in situ formed particles of an intermetallic Mg<sub>x</sub>(M) where M is any element on the periodic table or any compound in a magnesium matrix and wherein M has a melting point that is greater than the melting point of Mg.

**DETAILED DESCRIPTION OF THE INVENTION**

The present invention is directed to a novel magnesium composite that can be used to form a castable, moldable, or extrudable component. The magnesium composite includes at least 50 wt. % magnesium. Generally, the magnesium composite includes over 50 wt. % magnesium and less than about 99.5 wt. % magnesium and all values and ranges therebetween. One or more additives are added to a magnesium or magnesium alloy to form the novel magnesium composite of the present invention. The one or more additives can be selected and used in quantities so that galvanically-

**16**

active intermetallic or insoluble precipitates form in the magnesium or magnesium alloy while the magnesium or magnesium alloy is in a molten state and/or during the cooling of the melt; however, this is not required. The one or more additives are added to the molten magnesium or magnesium alloy at a temperature that is less than the melting point of the one or more additives. During the process of mixing the one or more additives in the molten magnesium or magnesium alloy, the one or more additives are not caused to fully melt in the molten magnesium or magnesium alloy. After the mixing process is completed, the molten magnesium or magnesium alloy and the one or more additives that are mixed in the molten magnesium or magnesium alloy are cooled to form a solid component. Such a formation in the melt is called in situ particle formation as illustrated in FIGS. 1-3. Such a process can be used to achieve a specific galvanic corrosion rate in the entire magnesium composite and/or along the grain boundaries of the magnesium composite. This feature results in the ability to control where the galvanically-active phases are located in the final casting, as well as the surface area ratio of the in situ phase to the matrix phase, which enables the use of lower cathode phase loadings as compared to a powder metallurgical or alloyed composite to achieve the same dissolution rates. The in situ formed galvanic additives can be used to enhance mechanical properties of the magnesium composite such as ductility, tensile strength, and/or shear strength. The final magnesium composite can also be enhanced by heat treatment as well as deformation processing (such as extrusion, forging, or rolling) to further improve the strength of the final composite over the as-cast material; however, this is not required. The deformation processing can be used to achieve strengthening of the magnesium composite by reducing the grain size of the magnesium composite. Further enhancements, such as traditional alloy heat treatments (such as solutionizing, aging and/or cold working) can be used to enable control of dissolution rates through precipitation of more or less galvanically-active phases within the alloy microstructure while improving mechanical properties; however, this is not required. Because galvanic corrosion is driven by both the electro potential between the anode and cathode phase, as well as the exposed surface area of the two phases, the rate of corrosion can also be controlled through adjustment of the in situ formed particles size, while not increasing or decreasing the volume or weight fraction of the addition, and/or by changing the volume/weight fraction without changing the particle size. Achievement of in situ particle size control can be achieved by mechanical agitation of the melt, ultrasonic processing of the melt, controlling cooling rates, and/or by performing heat treatments. In situ particle size can also or alternatively be modified by secondary processing such as rolling, forging, extrusion and/or other deformation techniques. A smaller particle size can be used to increase the dissolution rate of the magnesium composite. An increase in the weight percent of the in situ formed particles or phases in the magnesium composite can also or alternatively be used to increase the dissolution rate of the magnesium composite. A phase diagram for forming in situ formed particles or phases in the magnesium composite is illustrated in FIG. 4.

In accordance with the present invention, a novel magnesium composite is produced by casting a magnesium metal or magnesium alloy with at least one component to form a galvanically-active phase with another component in the chemistry that forms a discrete phase that is insoluble at the use temperature of the dissolvable component. The in

## US 10,329,653 B2

17

situ formed particles and phases have a different galvanic potential from the remaining magnesium metal or magnesium alloy. The in situ formed particles or phases are uniformly dispersed through the matrix metal or metal alloy using techniques such as thixomolding, stir casting, mechanical agitation, chemical agitation, electrowetting, ultrasonic dispersion, and/or combinations of these methods. Due to the particles being formed in situ to the melt, such particles generally have excellent wetting to the matrix phase and can be found at grain boundaries or as continuous dendritic phases throughout the component depending on alloy composition and the phase diagram. Because the alloys form galvanic intermetallic particles where the intermetallic phase is insoluble to the matrix at use temperatures, once the material is below the solidus temperature, no further dispersing or size control is necessary in the component. This feature also allows for further grain refinement of the final alloy through traditional deformation processing to increase tensile strength, elongation to failure, and other properties in the alloy system that are not achievable without the use of insoluble particle additions. Because the ratio of in situ formed phases in the material is generally constant and the grain boundary to grain surface area is typically consistent even after deformation processing and heat treatment of the composite, the corrosion rate of such composites remains very similar after mechanical processing.

## EXAMPLE I

An AZ91D magnesium alloy having 9 wt. % aluminum, 1 wt. % zinc and 90 wt. % magnesium was melted to above 800° C. and at least 200° C. below the melting point of nickel. About 7 wt. % of nickel was added to the melt and dispersed. The melt was cast into a steel mold. The cast material exhibited a tensile strength of about 14 ksi, an elongation of about 3%, and shear strength of 11 ksi. The cast material dissolved at a rate of about 75 mg/cm<sup>2</sup>-min in a 3% KCl solution at 90° C. The material dissolved at a rate of 1 mg/cm<sup>2</sup>-hr in a 3% KCl solution at 21° C. The material dissolved at a rate of 325 mg/cm<sup>2</sup>-hr. in a 3% KCl solution at 90° C.

## EXAMPLE 2

The composite in Example 1 was subjected to extrusion with an 11:1 reduction area. The material exhibited a tensile yield strength of 45 ksi, an Ultimate tensile strength of 50 ksi and an elongation to failure of 8%. The material has a dissolve rate of 0.8 mg/cm<sup>2</sup>-min. in a 3% KCl solution at 20° C. The material dissolved at a rate of 100 mg/cm<sup>2</sup>-hr. in a 3% KCl solution at 90° C.

## EXAMPLE 3

The alloy in Example 2 was subjected to an artificial T5 age treatment of 16 hours from 100° C.-200° C. The alloy exhibited a tensile strength of 48 ksi and elongation to failure of 5% and a shear strength of 25 ksi. The material dissolved at a rate of 110 mg/cm<sup>2</sup>-hr. in 3% KCl solution at 90° C. and 1 mg/cm<sup>2</sup>-hr. in 3% KCl solution at 20° C.

## EXAMPLE 4

The alloy in Example 1 was subjected to a solutionizing treatment T4 of 18 hours from 400° C.-500° C. and then an artificial T6 aging treatment of 16 hours from 100° C.-200° C. The alloy exhibited a tensile strength of 34 ksi and

18

elongation to failure of 11% and a shear strength of 18 ksi. The material dissolved at a rate of 84 mg/cm<sup>2</sup>-hr. in 3% KCl solution at 90° C. and 0.8 mg/cm<sup>2</sup>-hr. in 3% KCl solution at 20° C.

## EXAMPLE 5

An AZ91D magnesium alloy having 9 wt. % aluminum, 1 wt. % zinc and 90 wt. % magnesium was melted to above 800° C. and at least 200° C. below the melting point of copper. About 10 wt. % of copper alloyed to the melt and dispersed. The melt was cast into a steel mold. The cast material exhibited a tensile yield strength of about 14 ksi, an elongation of about 3%, and shear strength of 11 ksi. The cast material dissolved at a rate of about 50 mg/cm<sup>2</sup>-hr. in a 3% KCl solution at 90° C. The material dissolved at a rate of 0.6 mg/cm<sup>2</sup>-hr. in a 3% KCl solution at 21° C.

## EXAMPLE 6

The alloy in Example 5 was subjected to an artificial T5 aging treatment of 16 hours from 100° C.-200° C. the alloy exhibited a tensile strength of 50 ksi and elongation to failure of 5% and a shear strength of 25 ksi. The material dissolved at a rate of 40 mg/cm<sup>2</sup>-hr. in 3% KCl solution at 90° C. and 0.5 mg/cm<sup>2</sup>-hr. in 3% KCl solution at 20° C.

It will thus be seen that the objects set forth above, among those made apparent from the preceding description, are efficiently attained, and since certain changes may be made in the constructions set forth without departing from the spirit and scope of the invention, it is intended that all matter contained in the above description and shown in the accompanying drawings shall be interpreted as illustrative and not in a limiting sense. The invention has been described with reference to preferred and alternate embodiments. Modifications and alterations will become apparent to those skilled in the art upon reading and understanding the detailed discussion of the invention provided herein. This invention is intended to include all such modifications and alterations insofar as they come within the scope of the present invention. It is also to be understood that the following claims are intended to cover all of the generic and specific features of the invention herein described and all statements of the scope of the invention, which, as a matter of language, might be said to fall there between. The invention has been described with reference to the preferred embodiments. These and other modifications of the preferred embodiments as well as other embodiments of the invention will be obvious from the disclosure herein, whereby the foregoing descriptive matter is to be interpreted merely as illustrative of the invention and not as a limitation. It is intended to include all such modifications and alterations insofar as they come within the scope of the appended claims.

## What is claimed:

1. A magnesium composite that includes in situ precipitation of galvanically-active intermetallic phases to enable controlled dissolution of said magnesium composite, said magnesium composite comprising a mixture of magnesium or a magnesium alloy and an additive material, said additive material having a greater melting point temperature than a solidus temperature of said magnesium, said additive material constituting about 0.05 wt. %-45 wt. % of said mixture, said additive material forming precipitant in said magnesium composite, said additive material includes one or more metals selected from the group consisting of copper, nickel,

## US 10,329,653 B2

19

iron, and cobalt, said magnesium composite has a dissolution rate of at least 5 mg/cm<sup>2</sup>/hr. in 3 wt. % KCl water mixture at 90° C.

2. The magnesium composite as defined in claim 1, wherein said magnesium alloy includes over 50 wt. % magnesium and one or more metals selected from the group consisting of aluminum, boron, bismuth, zinc, zirconium, and manganese.

3. The magnesium composite as defined in claim 1, wherein said magnesium alloy includes over 50 wt. % magnesium and one or more metals selected from the group consisting of aluminum in an amount of about 0.5-10 wt. %, zinc in amount of about 0.1-3 wt. %, zirconium in an amount of about 0.01-1 wt. %, manganese in an amount of about 0.15-2 wt. %, boron in amount of about 0.0002-0.04 wt. %, and bismuth in amount of about 0.4-0.7 wt. %.

4. The magnesium composite as defined in claim 1, wherein said additive material includes nickel, said nickel constitutes about 0.05-35 wt. % of said magnesium composite, said nickel forms galvanically-active in situ precipitate in said magnesium composite.

5. The magnesium composite as defined in claim 1, wherein said additive material includes copper, said copper constitutes about 0.05-35 wt. % of said magnesium composite, said copper forms galvanically-active in situ precipitate in said magnesium composite.

6. The magnesium composite as defined in claim 1, wherein said additive material includes cobalt, said cobalt constitutes about 0.05-35 wt. % of said magnesium composite, said cobalt forms galvanically-active in situ precipitate in said magnesium composite.

7. The magnesium composite as defined in claim 1, where said magnesium composite is subjected to a deformation processing to reduce grain size of said magnesium composite, increase tensile yield strength of said magnesium composite, increase elongation of said magnesium composite, or combinations thereof.

8. The magnesium composite as defined in claim 1, wherein said magnesium composite is subjected to a surface treatment to improve a surface hardness of said magnesium composite, said surface treatment including peening, heat treatment, aluminizing, or combinations thereof.

9. The magnesium composite as defined in claim 1, wherein a dissolution rate of said magnesium composite is about 5-325 mg/cm<sup>2</sup>/hr. in 3 wt. % KCl water mixture at 90° C.

10. The magnesium composite as defined in claim 1, wherein a dissolution rate of said magnesium composite is controlled by an amount and size of said in situ formed galvanically-active particles whereby smaller average sized particles of said in situ formed galvanically-active particles, a greater weight percent of said in situ formed galvanically-active particles in said magnesium composite, or combinations thereof increases said dissolution rate of said magnesium composite.

11. The magnesium composite as defined in claim 1, wherein said additive material is a metal or metal alloy.

12. A magnesium composite that includes in situ precipitation of galvanically-active intermetallic phases to enable controlled dissolution of said magnesium composite comprising a mixture of a magnesium or a magnesium alloy and an additive material, said additive material having a greater melting point temperature than a solidus temperature of said magnesium, said composite including greater than 50 wt. % magnesium, said additive material constituting about 0.05-45 wt. % of said magnesium composite, said additive material having a melting point temperature that is 100° C.

20

greater than a melting temperature of said magnesium or magnesium alloy, said additive material including one or more metals selected from the group consisting of copper, nickel, cobalt, titanium, and iron, at least a portion of said additive material remaining unalloyed additive material, said magnesium composite including in situ precipitation of galvanically-active intermetallic phases that includes said unalloyed additive material, said magnesium composite has a dissolution rate of at least 5 mg/cm<sup>2</sup>/hr. in 3 wt. % KCl water mixture at 90° C.

13. The magnesium composite as defined in claim 12, wherein said additive material is added to said magnesium or magnesium alloy while said magnesium or magnesium alloy is at a temperature that is above said solidus temperature of said magnesium and a temperature that is less than a melting point of said additive material to form said mixture.

14. The magnesium composite as defined in claim 13, wherein said magnesium alloy includes over 50 wt. % magnesium and one or more metals selected from the group consisting of aluminum in an amount of about 0.5-10 wt. %, zinc in amount of about 0.1-3 wt. %, zirconium in an amount of about 0.01-1 wt. %, manganese in an amount of about 0.15-2 wt. %, boron in amount of about 0.0002-0.04 wt. %, and bismuth in an amount of about 0.4-0.7 wt. %.

15. The magnesium composite as defined in claim 14, wherein said additive material includes nickel, said nickel constitutes about 0.05-35 wt. % of said magnesium composite, said nickel forms galvanically-active in situ precipitate in said magnesium composite.

16. The magnesium composite as defined in claim 15, wherein said additive material includes nickel, said nickel constitutes about 3-7 wt. % of said magnesium composite.

17. The magnesium composite as defined in claim 15, wherein said additive material includes nickel, said nickel constitutes about 7-10 wt. % of said magnesium composite.

18. The magnesium composite as defined in claim 15, wherein a dissolution rate of said magnesium composite is at least 45 mg/cm<sup>2</sup>/hr. in 3 wt. % KCl water mixture at 90° C. and up to 325 mg/cm<sup>2</sup>/hr. in 3 wt. % KCl water mixture at 90° C.

19. The magnesium composite as defined in claim 12, wherein said magnesium alloy includes over 50 wt. % magnesium and one or more metals selected from the group consisting of aluminum in an amount of about 0.5-10 wt. %, zinc in amount of about 0.1-6 wt. %, zirconium in an amount of about 0.01-3 wt. %, manganese in an amount of about 0.15-2 wt. %, boron in amount of about 0.0002-0.04 wt. %, and bismuth in an amount of about 0.4-0.7 wt. %.

20. The magnesium composite as defined in claim 12, said additive material includes nickel, said nickel constitutes about 0.05-35 wt. % of said magnesium composite, said nickel forms galvanically-active in situ precipitate in said magnesium composite.

21. The magnesium composite as defined in claim 20, wherein said additive material includes nickel, said nickel constitutes about 3-7 wt. % of said magnesium composite.

22. The magnesium composite as defined in claim 20, wherein said additive material includes nickel, said nickel constitutes about 7-10 wt. % of said magnesium composite.

23. The magnesium composite as defined in claim 12, wherein a dissolution rate of said magnesium composite is at least 45 mg/cm<sup>2</sup>/hr. in 3 wt. % KCl water mixture at 90° C. and up to 325 mg/cm<sup>2</sup>/hr. in 3 wt. % KCl water mixture at 90° C.

24. The magnesium composite as defined in claim 12, wherein said additive material is a metal or metal alloy.

## US 10,329,653 B2

21

**25.** A dissolvable magnesium alloy composite for use in a ball or other tool component in a well drilling or completion operation, said dissolvable magnesium alloy composite comprising at least 85 wt. % magnesium; one or more metals selected from the group consisting of 0.5-10 wt. % aluminum, 0.05-6 wt. % zinc, 0.01-3 wt. % zirconium, and 0.15-2 wt. % manganese; and about 0.05-45 wt. % of a secondary metal to form a galvanically-active intermetallic particle that promotes corrosion of said dissolvable magnesium alloy composite, said secondary metal including one or more metals selected from the group consisting of copper, nickel, cobalt, titanium and iron, said magnesium alloy composite has a dissolution rate of at least 5 mg/cm<sup>2</sup>/hr. in 3 wt. % KCl water mixture at 90° C.

**26.** The dissolvable magnesium alloy composite as defined in claim **25**, wherein a dissolution rate of said magnesium alloy composite is 100-325 mg/cm<sup>2</sup>/hr. in 3 wt. % KCl water mixture at 90° C.

**27.** The dissolvable magnesium composite as defined in claim **25**, wherein said secondary metal includes nickel, said nickel forms galvanically-active in situ precipitate in said magnesium composite.

**28.** The dissolvable magnesium alloy composite as defined in claim **25**, wherein said additive material is a metal or metal alloy.

**29.** A dissolvable magnesium alloy composite for use in a ball or other tool component in a well drilling or completion operation, said dissolvable magnesium alloy composite comprising 60-95 wt. % magnesium; 0.01-1 wt. % zirconium; and about 0.05-45 wt. % of a secondary metal to form a galvanically-active intermetallic particle that promotes corrosion of said dissolvable magnesium alloy composite, said secondary metal including one or more metals selected from the group consisting of copper, nickel, cobalt, titanium and iron, said magnesium alloy composite has a dissolution rate of at least 5 mg/cm<sup>2</sup>/hr. in 3 wt. % KCl water mixture at 90° C.

**30.** The dissolvable magnesium alloy composite as defined in claim **29**, wherein a dissolution rate of said magnesium alloy composite is 5-325 mg/cm<sup>2</sup>/hr. in 3 wt. % KCl water mixture at 90° C.

**31.** The dissolvable magnesium composite as defined in claim **29**, wherein said secondary metal includes nickel, said nickel forms galvanically-active in situ precipitate in said magnesium composite.

**32.** The dissolvable magnesium alloy composite as defined in claim **29**, wherein said additive material is a metal or metal alloy.

**33.** A dissolvable magnesium alloy composite for use in a ball or other tool component in a well drilling or completion operation, said dissolvable magnesium alloy composite comprising 60-95 wt. % magnesium; 0.5-10 wt. % aluminum; 0.05-6 wt. % zinc; 0.15-2 wt. % manganese; and about 0.05-45 wt. % of a secondary metal to form a galvanically-active intermetallic particle that promotes corrosion of said dissolvable magnesium alloy composite, said secondary metal including one or more metals selected from the group consisting of copper, nickel, cobalt, titanium and iron, said magnesium alloy composite has a dissolution rate of at least 5 mg/cm<sup>2</sup>/hr. in 3 wt. % KCl water mixture at 90° C.

**34.** The dissolvable magnesium alloy composite as defined in claim **33**, wherein a dissolution rate of said magnesium alloy composite is 5-325 mg/cm<sup>2</sup>/hr. in 3 wt. % KCl water mixture at 90° C.

22

**35.** The dissolvable magnesium composite as defined in claim **33**, wherein said secondary metal includes nickel, said nickel forms galvanically-active in situ precipitate in said magnesium composite.

**36.** The dissolvable magnesium alloy composite as defined in claim **33**, wherein said additive material is a metal or metal alloy.

**37.** A dissolvable magnesium alloy composite for use in a ball or other tool component in a well drilling or completion operation, said dissolvable magnesium alloy composite comprising 60-95 wt. % magnesium; 0.05-6 wt. % zinc; 0.01-1 wt. % zirconium; and about 0.05-45 wt. % of a secondary metal to form a galvanically-active intermetallic particle that promotes corrosion of said dissolvable magnesium alloy composite, said secondary metal including one or more metals selected from the group consisting of copper, nickel, cobalt, titanium and iron, said magnesium alloy composite has a dissolution rate of at least 5 mg/cm<sup>2</sup>/hr. in 3 wt. % KCl water mixture at 90° C.

**38.** The dissolvable magnesium alloy composite as defined in claim **37**, wherein a dissolution rate of said magnesium alloy composite is 5-325 mg/cm<sup>2</sup>/hr. in 3 wt. % KCl water mixture at 90° C.

**39.** The dissolvable magnesium composite as defined in claim **37**, wherein said secondary metal includes nickel, said nickel forms galvanically-active in situ precipitate in said magnesium composite.

**40.** The dissolvable magnesium alloy composite as defined in claim **37**, wherein said additive material is a metal or metal alloy.

**41.** A dissolvable magnesium alloy composite for use in a ball or other tool component in a well drilling or completion operation, said dissolvable magnesium alloy composite comprising over 50 wt. % magnesium; one or more metals selected from the group consisting of 0.5-10 wt. % aluminum, 0.1-2 wt. % zinc, 0.01-1 wt. % zirconium, and 0.15-2 wt. % manganese; and about 0.05-45 wt. % of a secondary metal to form a galvanically-active intermetallic particle that promotes corrosion of said dissolvable magnesium alloy composite, said secondary metal including one or more metals selected from the group consisting of copper, nickel and cobalt, said magnesium alloy composite has a dissolution rate of at least 5 mg/cm<sup>2</sup>/hr. in 3 wt. % KCl water mixture at 90° C.

**42.** The dissolvable magnesium alloy composite as defined in claim **41**, wherein a dissolution rate of said magnesium alloy composite is 5-325 mg/cm<sup>2</sup>/hr. in 3 wt. % KCl water mixture at 90° C.

**43.** The dissolvable magnesium composite as defined in claim **41**, wherein said secondary metal includes nickel, said nickel forms galvanically-active in situ precipitate in said magnesium composite.

**44.** The dissolvable magnesium alloy composite as defined in claim **41**, wherein said additive material is a metal or metal alloy.

**45.** A dissolvable magnesium alloy composite for use in a ball or other tool component in a well drilling or completion operation, said dissolvable magnesium alloy composite comprising over 50 wt. % magnesium; one or more metals selected from the group consisting of 0.1-3 wt. % zinc, 0.01-1 wt. % zirconium, 0.05-1 wt. % manganese, 0.0002-0.04 wt. % boron, and 0.4-0.7 wt. % bismuth; and about 0.05-45 wt. % of a secondary metal to form a galvanically-active intermetallic particle that promotes corrosion of said dissolvable magnesium alloy composite, said secondary metal including one or more metals selected from the group consisting of copper, nickel, and cobalt, said magnesium

US 10,329,653 B2

23

alloy composite has a dissolution rate of at least 5 mg/cm<sup>2</sup>/hr. in 3 wt. % KCl water mixture at 90° C.

**46.** The dissolvable magnesium alloy composite as defined in claim **45**, wherein a dissolution rate of said magnesium alloy composite is 5-325 mg/cm<sup>2</sup>/hr. in 3 wt. % KCl water mixture at 90° C.

**47.** The dissolvable magnesium composite as defined in claim **45**, wherein said secondary metal includes nickel, said nickel forms galvanically-active in situ precipitate in said magnesium composite.

**48.** The dissolvable magnesium alloy composite as defined in claim **45**, wherein said additive material is a metal or metal alloy.

**49.** A magnesium composite that includes in situ precipitation of galvanically-active intermetallic phases to enable controlled dissolution of said magnesium composite, said magnesium composite comprising a mixture of magnesium or a magnesium alloy and an additive material, said additive material constituting about 0.05-45 wt. % of said mixture, said additive material includes one or more metals selected from the group consisting of copper, nickel, titanium, iron, and cobalt, said magnesium composite including in situ precipitation of galvanically-active intermetallic phases that include said additive material, said additive material located in sufficient quantities in said galvanically-active intermetallic phases so as to obtain a composition and morphology of said galvanically-active intermetallic phases such that a galvanic corrosion rate along said galvanically-active intermetallic phases causes said magnesium composite to have a dissolution rate of at least at least 5 mg/cm<sup>2</sup>/hr. in 3 wt. % KCl water mixture at 90° C.

**50.** The magnesium composite as defined in claim **49**, wherein said additive material includes one or more metals selected from the group consisting of copper, nickel, and cobalt.

**51.** The magnesium composite as defined in claim **49**, wherein said additive material includes particles having an average particle diameter size of about 0.1-500 microns.

**52.** The magnesium composite as defined in claim **49**, wherein said magnesium alloy includes over 50 wt. % magnesium, and one or more metals selected from the group consisting of aluminum, boron, bismuth, zinc, zirconium, and manganese.

**53.** The magnesium composite as defined in claim **49**, wherein said magnesium alloy includes over 50 wt. % magnesium, and one or more metals selected from the group consisting of aluminum in an amount of about 0.5-10 wt. %, zinc in an amount of about 0.1-6 wt. %, zirconium in an amount of about 0.01-3 wt. %, manganese in an amount of about 0.15-2 wt. %, boron in an amount of about 0.0002-0.04 wt. %, and bismuth in amount of about 0.4-0.7 wt. %.

**54.** The magnesium composite as defined in claim **49**, wherein said magnesium alloy includes over 50 wt. % magnesium, and one or more metals selected from the group consisting of aluminum in an amount of about 0.5-10 wt. %, zinc in an amount of about 0.1-3 wt. %, zirconium in an amount of about 0.01-1 wt. %, manganese in an amount of about 0.15-2 wt. %, boron in an amount of about 0.0002-0.04 wt. %, and bismuth in an amount of about 0.4-0.7 wt. %.

**55.** The magnesium composite as defined in claim **49**, wherein said magnesium alloy includes at least 85 wt. % magnesium, and one or more metals selected from the group consisting of 0.5-10 wt. % aluminum, 0.05-6 wt. % zinc, 0.01-3 wt. % zirconium, and 0.15-2 wt. % manganese.

24

**56.** The magnesium composite as defined in claim **49**, wherein said magnesium alloy includes 60-95 wt. % magnesium and 0.01-1 wt. % zirconium.

**57.** The magnesium composite as defined in claim **56**, wherein said magnesium alloy further includes 0.05-6 wt. % zinc.

**58.** The magnesium composite as defined in claim **49**, wherein said magnesium alloy includes 60-95 wt. % magnesium, 0.5-10 wt. % aluminum, 0.05-6 wt. % zinc, and 0.15-2 wt. % manganese.

**59.** The magnesium composite as defined in claim **49**, wherein said magnesium alloy includes over 50 wt. % magnesium and one or more metals selected from the group consisting of 0.5-10 wt. % aluminum, 0.1-2 wt. % zinc, 0.01-1 wt. % zirconium, and 0.15-2 wt. % manganese.

**60.** The magnesium composite as defined in claim **49**, wherein said magnesium alloy includes over 50 wt. % magnesium, and one or more metals selected from the group consisting of 0.1-3 wt. % zinc, 0.01-1 wt. % zirconium, 0.05-1 wt. % manganese, 0.0002-0.04 wt. % boron, and 0.4-0.7 wt. % bismuth.

**61.** The magnesium composite as defined in claim **49**, wherein said additive material includes nickel, said nickel constitutes about 0.05-35 wt. % of said magnesium composite, said nickel forms galvanically-active in situ precipitate in said magnesium composite.

**62.** The magnesium composite as defined in claim **61**, wherein said nickel constitutes about 3-7 wt. % of said magnesium composite.

**63.** The magnesium composite as defined in claim **61**, wherein said nickel constitutes about 7-10 wt. % of said magnesium composite.

**64.** The magnesium composite as defined in claim **49**, wherein said additive material includes copper, said copper constitutes about 0.05-35 wt. % of said magnesium composite, said copper forms the galvanically-active in situ precipitate in said magnesium composite.

**65.** The magnesium composite as defined in claim **49**, wherein said additive material includes cobalt, said cobalt constitutes about 0.05-35 wt. % of said magnesium composite, said cobalt forms the galvanically-active in situ precipitate in said magnesium composite.

**66.** The magnesium composite as defined in claim **49**, wherein a dissolution rate of said magnesium composite is about 5-325 mg/cm<sup>2</sup>/hr. in 3 wt. % KCl water mixture at 90° C.

**67.** The magnesium composite as defined in claim **49**, wherein a dissolution rate of said magnesium composite is at least 45 mg/cm<sup>2</sup>/hr. in 3 wt. % KCl water mixture at 90° C. and up to 325 mg/cm<sup>2</sup>/hr. in 3 wt. % KCl water mixture at 90° C.

**68.** The magnesium composite as defined in claim **49**, wherein a dissolution rate of said magnesium composite is up to 1 mg/cm<sup>2</sup>/hr. in 3 wt. % KCl water mixture at 21° C.

**69.** The magnesium composite as defined in claim **49**, wherein said additive material has a melting point temperature that is 100° C. greater than a melting temperature of said magnesium or magnesium alloy.

**70.** The magnesium composite as defined in claim **49**, wherein said magnesium composite is at least partially included in a down hole well component, said down hole well component including one or more components selected from the group consisting of a sleeve, frac ball, hydraulic actuating tooling, tube, valve, valve component, or plug.

**71.** The magnesium composite as defined in claim **49**, wherein at least a portion of said additive material remains unalloyed additive material in said magnesium composite.

US 10,329,653 B2

25

72. The magnesium composite as defined in claim 49, wherein said additive material is a metal or metal alloy.

73. A dissolvable magnesium composite for use in a ball or other tool component in a well drilling or completion operation, said dissolvable magnesium composite includes in situ precipitation of galvanically-active intermetallic phases to enable controlled dissolution of said magnesium composite, said magnesium composite comprising a mixture of magnesium or a magnesium alloy and an additive material, said additive material constituting about 0.05 wt. % of said mixture, said additive material is a metal or metal alloy, said additive material includes one or more metals selected from the group consisting of copper, nickel, titanium, iron, silicon, and cobalt, said magnesium composite including in situ precipitation of galvanically-active intermetallic phases that include said additive material, said additive material located in sufficient quantities in said galvanically-active intermetallic phases so as to obtain a composition and morphology of said galvanically-active intermetallic phases such that a galvanic corrosion rate along said galvanically-active intermetallic phases causes said magnesium composite to have a dissolution rate of at least at least 5 mg/cm<sup>2</sup>/hr. in 3 wt. % KCl water mixture at 90° C.

74. A dissolvable magnesium composite for use in a ball or other tool component in a well drilling or completion operation, said dissolvable magnesium composite includes in situ precipitation of galvanically-active intermetallic

26

phases to enable controlled dissolution of said magnesium composite, said magnesium composite comprising a mixture of magnesium or a magnesium alloy and an additive material, said additive material constituting at least 0.1 wt. % of said mixture, said magnesium in said magnesium composite constituting at least 85 wt. %, said additive material is a metal material selected from the group consisting of copper, nickel and cobalt, said magnesium composite including in situ precipitation of galvanically-active intermetallic phases that include said additive material, said magnesium composite has a dissolution rate of 84-325 mg/cm<sup>2</sup>/hr. in 3 wt. % KCl water mixture at 90° C.

75. The magnesium composite as defined in claim 74, wherein said magnesium alloy is an AZ91D magnesium alloy that includes aluminum and zinc.

76. The magnesium composite as defined in claim 74, wherein said additive material includes nickel, a content of said nickel in said magnesium composite is at least 0.3 wt. %.

77. The magnesium composite as defined in claim 74, wherein said additive material includes nickel, a content of said nickel in said magnesium composite is at least 7 wt. %.

78. The magnesium composite as defined in claim 74, wherein said additive material includes nickel, a content of said nickel in said magnesium composite is at least 10 wt. %.

\* \* \* \* \*